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# Characterization of the ORNL MVST Waste Tanks After Transfer of Sludge from BVEST, GAAT, and OHF Tanks

J. M. Keller J. M. Giaquinto



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#### Chemical and Analytical Sciences Division

# Characterization of the ORNL MVST Waste Tanks After Transfer of Sludge from BVEST, GAAT, and OHF Tanks

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#### ABBREVIATIONS AND ACRONYMS

ALARA As Low As Reasonably Achievable

CAO Carlsbad Area Office

CASD Chemical and Analytical Sciences Division

CVAA Cold Vapor Atomic Absorption

DQO Data Quality Objective

EPA Environmental Protection Agency

GC/MS Gas Chromatography/Mass Spectrometry

GC Gas Chromatography

GFAA Graphite Furnace Atomic Absorption

IC Ion Chromatography

ICP Inductively Coupled Plasma

ICP-AES Inductively Coupled Plasma - Atomic Emission Spectroscopy

ICP-MS Inductively Coupled Plasma - Mass Spectrometry

IDL Instrument Detection Limit LCS Laboratory Control Sample

LLLW Liquid Low-Level Waste

MDL Method Detection Limit

MS Matrix Spike

MSD Matrix Spike Duplicate
MVST Melton Valley Storage Tanks

NTS Nevada Test Site

ORNL Oak Ridge National Laboratory

QA Quality Assurance

QAPjP Quality Assurance Project Plan QAPP Quality Assurance Program Plan

QC Quality Control

RCRA Resource Conservation and Recovery Act

RMAL Radioactive Materials Analytical Laboratory (Building 2026)

TC Total Carbon

TCLP Toxicity Characteristic Leaching Procedure

TDS Total Dissolved Solids

TIMS Thermal Ionization Mass Spectrometry

TOC Total Organic Carbon

TRU Transuranic

TWCP Transuranic Waste Characterization Program

WAC Waste Acceptance Criteria WIPP Waste Isolation Pilot Plant

#### **EXECUTIVE SUMMARY**

Over the last several years most of the sludge and liquid from the Liquid Low-Level Waste (LLLW) tanks at ORNL has been transferred and consolidated in the Melton Valley Storage Tanks (MVST). The contents of the MVST tanks at the time the sludge samples were collected for this report included the original inventory in the MVSTs along with the sludge and liquid from the Bethel Valley Evaporator Service Tanks (BVEST), Old Hydrofracture (OHF) tanks, and most of the Gunite and Associated Tanks (GAAT). During the spring and summer of 2000 the MVST composite sludge was sampled and characterized to validate the radiochemical content and to ensure regulatory compliance. This report only discusses the analytical characterization of the sludge from the MVST waste tanks (except for W-29 and W-30).

The isotopic data presented in this report supports the position that fissile isotopes of uranium (<sup>233</sup>U and <sup>235</sup>U) and plutonium (<sup>239</sup>Pu and <sup>241</sup>Pu) were "denatured" as required by the administrative controls stated in the ORNL LLLW waste acceptance criteria (WAC). In general, the MVST sludge was found to be hazardous by RCRA characteristics based on total analysis of chromium, mercury, and lead. Also, the alpha activity due to transuranic isotopes was well above the 100 nCi/g limit for TRU waste. The characteristics of the MVST sludge relative to the WIPP WAC limits for fissile gram equivalent, plutonium equivalent activity, and thermal power from decay heat, were estimated from the data in this report and found to be far below the upper boundary for any of the remote-handled transuranic waste (RH-TRU) requirements for disposal of the waste in WIPP.



# Characterization of the MVST Waste Tanks Located at ORNL After Transfer of Sludge from BVEST, GAAT, and OHF Tanks

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#### 1.0 Introduction

The active ORNL Liquid Low Level Waste (LLLW) system consists of the set of waste tanks summarized in Table 1. As indicated in Table 1, this report only discusses the analytical characterization data for the Melton Valley Storage Tanks (MVST) (except for W-29 and W-30). The characterization data summarized in this report was needed to verify the current composition of the sludge present after the transfer of sludge from the Bethel Valley Evaporator Service Tanks (BVEST), Gunite and Associated Tanks (GAAT), and the Old Hydrofracture (OHF) tanks. The data in this report can also be used to address waste processing options, support the performance assessment (PA) requirements for the Waste Isolation Pilot Plant (WIPP), evaluate the waste characteristics with respect to the waste acceptance criteria (WAC) for WIPP and Nevada Test Site (NTS), address criticality concerns, and to meet DOT requirements for transporting the waste.

The samples and data collected for this project were performed during May and June of 2000. The level of quality assurance approximates that required for regulatory measurements with the understanding that, when needed, sample size requirements were reduced, and steps were taken to reduce sample handling to ensure radiation exposures were as-low-as-reasonably-achievable (ALARA). Some procedure modifications were required to handle chemical matrix problems due to the high levels of sodium nitrate, uranium, and thorium present. Any deviations from procedures or problems observed with the tank samples were documented in the data files maintained by the laboratory. The regulatory holding time requirements for mercury and the organic measurements were complied with unless noted differently in the data tables. The Quality Control (QC) Acceptance Criteria for measurement used on this project are summarized in Appendix B.

Table 1 Summary of MVST Tanks in the ORNL LLLW System

	Data Presented in this report			
Tanks	Liquid	Sludge		
MVST TANKS				
W-24	none	✓		
W-25	none	✓		
W-26	none	✓		
W-27	none	✓		
W-28	none	✓		
W-29	none	none		
W-30	none	none		
W-31	none	✓		

## 2.0 Sample Collection Activities

A detailed description on the background, operation of the LLLW system, and the sample collection techniques has been presented in previous reports and will not be discussed here (see Sections 2 and 3 of Reference 1). The staff from the Liquid and Gaseous Waste Operations (LGWO) provided all sample collection support and delivered the samples to the analytical laboratory. The tank location for sludge samples collected are illustrated in Appendix A. The documentation for chain-of-custody was prepared, maintained for each sample collected, and stored with the data files by the analytical laboratory.

## 3.0 Analytical Methodology

The information and data collected from these studies are used to support various activities. The activities include demonstration of regulatory compliance, measurements to support processing options, and to meet data needs for risk assessments and other safety related assessments such as

criticality. Standardized analytical procedures are used to the extent possible to ensure broad acceptance of the data generated. Unless stated otherwise, the U.S. Environmental Protection Agency (EPA) methods are used for the analyses of constituents listed as hazardous under the Resource Conservation and Recovery Act (RCRA), which includes all the inorganic and organic measurements presented in this report. In general, the EPA Guidance Manual, *Test Methods for Evaluating Solid Waste*<sup>2</sup> (SW-846), is used for inorganic and organic methods. Some modifications of the standard procedures are necessary to handle the high radiation levels and the high salt/solids content. Some procedure modifications are required to generate valid data, these changes were usually needed to correct for chemical or other matrix related interferences. All deviations from the standard procedures are documented in the raw data files and can be provided upon request to data users.

#### 3.1 <u>Sample Preparation</u>

The aqueous supernatant samples from the waste tanks were filtered or centrifuged to remove suspended particles. The clarified liquids were then digested by the SW-846 Method 3015, *Microwave Assisted Acid Digestion of Aqueous Samples and Extracts*. This sample preparation for aqueous samples was then used for all subsequent metal analyses by ICP-AES and GFAA, and most of the radiochemical analyses. Based upon results from a collaborative study<sup>3</sup> with Argonne National Laboratory - East (ANL-E), Method 3015/3051 demonstrated excellent recovery for mercury and was used to prepare tank samples for mercury determination.

The primary method for digesting the sludge samples was SW-846 Method 3051, *Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils*. This sample preparation is considered to be a total digestion for metals and radionuclides by regulatory agencies and yields good results for most metals and radionuclides of interest. This digestion gave poor performance on two of the metals of interest, silver and silicon. Although nitric acid is excellent for dissolving silver compounds, there is usually enough chloride present in waste samples to form an insoluble silver chloride (AgCl) precipitate. If the chloride concentration is increased sufficiently, a silver chloride complex (AgCl<sub>3</sub>-<sup>2</sup>) forms which is soluble in the aqueous environment. Improved matrix

spike recovery and defensible data for silver were obtained using a separate sample digestion discussed later in this report.

If the total silicon content in the sludge must be known to develop waste treatment options such as vitrification, another sample digestion is required. A simple nitric acid treatment will not dissolve most siliceous materials. The SW-846 Method 3052, Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices, provides the necessary digestion chemistry to yield good silicon data. Sludge samples were prepared for measurement of total silicon, by taking approximately 0.5 g of sludge and mixing with 7 mL of concentrated nitric acid and 3 mL of hydrofluoric acid in a fluorocarbon microwave vessel. The samples were digested for 10 minutes at 95% full power (570 watts) and then cooled to room temperature. The acid solution was then treated with excess boric acid and heated to 80°C for ten minutes to complex any free fluoride. This digestion mixture is cooled, filtered into a 50 mL volumetric flask, and diluted to volume with ASTM Type II water. Care must be exercised to ensure the digestion solution is cooled to room temperature prior to opening the sealed microwave vessel or there may be a significant loss of the volatile SiF<sub>4</sub>. The free fluoride is complexed with the boron to protect the sample introduction system to the ICP-AES and to prevent a high silicon background from the instrument glassware. This sample digestion with hydrofluoric acid should not be used for radiochemical measurements, especially for measurement of lanthanides or actinides.

Most of the metal and radionuclide data presented in this report are based upon a Method 3051 digestion with approximately a 0.5 gram sludge sample and 10 mL of concentrated nitric acid. After the microwave digestion is completed and the solution cooled to room temperature, the sample is filtered into a volumetric flask and diluted to 50 mL with ASTM Type II water or better. To ensure valid silver and antimony data, samples were digested in a similar manner except the 10 mL of nitric acid was replaced with 6 mL of concentrated nitric acid plus 4 mL of concentrated hydrochloric acid. Any residue remaining after the nitric acid or nitric-hydrochloric acid digestion consisted of mostly SiO<sub>2</sub> and was discarded.

#### 3.2 Metal Analysis

Three analytical measurement methods were used to determine all of the metals included in this report. Most of the metals are first determined by SW-846 Method 6010A, *Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES)*. There are several elements of interest for which the ICP-AES has insufficient detection limits, and these elements must be determined by Method 7000A, *Atomic Absorption Methods*. The Radioactive Materials Analytical Laboratory (RMAL) uses a Graphite Furnace Atomic Absorption (GFAA) Spectrometer for elements that require better sensitivity. The elements that usually require GFAA were antimony (Method 7041), arsenic (Method 7060A), lead (Method 7421), selenium (Method 7740), and thallium (Method 7841). All the mercury measurements are done by either Method 7470A, *Mercury in Liquid Waste (Manual Cold-Vapor Technique)*, or Method 7471A, *Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)*. The samples discussed in this report were prepared for mercury analysis by the microwave technique discussed in section 3.1, the sample preparation specified in the mercury methods (7470A and 7471A) were not used.

The level of radioactivity in most LLLW tank samples required that the analytical systems used for metal measurements be modified for operation in a radiochemical hood or glove box. Custom instrument configurations are necessary to ensure contamination control and worker safety. All work was performed in radiochemical laboratories which are operated under strict radiation protection programs, with the use of protective clothing and routine contamination monitoring. Both an ICP-AES system and a GFAA system can generate dry, dusty particles which are difficult to contain and are highly hazardous when radioactive. A detailed description of the RMAL setup for these instruments is given in Appendix B of Reference 1.

The instrument detection limits (IDL) for various metals with undiluted aqueous samples are listed in data tables along with the results. For sludge samples, these detection limits must be increased by a factor that represents the dilution that results from the sample preparation. For all the MVST sludge samples, approximately 0.5 g of sample was digested and then diluted to 50 mL which results in about a 100 fold dilution for the sample, and thus a 100 fold increase in the detection limits.

The analytical error for the metal measurements depends upon the analytical method, the concentration level, and the chemical matrix. Inductively-coupled plasma-atomic emission spectroscopy (ICP-AES) and inductively coupled plasma-mass spectrometry (ICP-MS) are both multi-element measurement techniques that are designed for the best average performance for all elements analyzed. In general, these measurement techniques are not optimized for any single element. The sample introduction system for ICP instruments adds additional variability due to changes in sample density, viscosity, and solids content between samples and/or calibration standards. Overall, the expected analytical error for ICP measurements range from  $\pm 4$ -6% at concentrations above 10 times the detection limit to  $\pm 20$ -50% near the detection limit. These error estimates are typical for both ICP-AES and ICP-MS measurements.

Graphite Furnace AA instruments are generally optimized for a specific element and usually provide lower detection limits and better precision. The expected analytical error for GFAA measurements range from 3-5% for concentrations greater than 10 times the detection limit to 20-40% near the detection limit. One advantage of GFAA analysis is that the measurements are normally well above the method's detection limits. The mercury measurements were done by Cold Vapor Atomic Absorption (CVAA), which is very selective and sensitive for mercury. The analytical errors for CVAA measurements are similar to GFAA work.

#### 3.3 Anion Analysis

The determination of the inorganic anions was needed for the development of process treatment options, to provide information to explain the distribution and chemical behaviors observed in the waste tanks, and to ensure the major chemical constituents were identified in the waste for which data was used to calculate the mass and charge balance for each sample. The common inorganic anions; including fluoride, chloride, bromide, phosphate, nitrate, nitrite, and sulfate; were measured by ion chromatography (IC) with a Dionex Model 4500i system. In addition, several water soluble organic acids were measured along with the inorganic anions. These organic acids were measured in their ionized form and included formate, acetate, citrate, and oxalate. Both the citrate and the oxalate can form strong complexes with many metals and change the solution chemistry of these

metals in the waste. The ion chromatography system used for measurements on these radioactive samples was configured such that the components that come into contact with radioactivity were isolated in a radiochemical hood for contamination control.

From past observations, the nitrate content dominates both the mass and charge balance calculations with sludge samples taken from the active LLLW tanks. There are many other anions present in the waste, some of which are measured directly by ion chromatography and others which can be estimated from the metal data such as chromate, dichromate, permanganate, and others. The carbonate is estimated from the total inorganic carbon measurement.

The anion measurement technique was ion chromatography. For simple water samples, without complex chemical matrix problems, the empirical analytical error for ion chromatography measurements ranges from 4-6% for concentrations above 10 times the detection limits to 20-40% near the detection limit. The measurement of anions present at concentration much lower (< 1/25) than other anionic species present may increase the overall error of the measurement.

#### 3.4 Radiochemical Analysis

The only standard radiochemical methods useful for radioactive waste characterization are EPA Method 600/900.0, *Gross Alpha and Beta Radioactivity in Drinking Water*, and EPA Method 600/901.1: *Gamma Emitting Radionuclides in Drinking Water*. The EPA Method 600/905.0, *Radioactive Strontium in Drinking Water*, gave poor performance with the chemical matrix found in ORNL LLLW supernatant and sludge samples. The EPA method for gross alpha/beta measurements uses gas-flow proportional counting. In general, this counting technique requires drying a sample at elevated temperatures onto a metal (usually stainless steel) plate, which resulted in the loss of cesium chloride from the MVST samples and yielded poor gross beta results. To avoid this problem, all gross beta measurements reported are based on measurements by liquid scintillation counting. Other than the gamma spectroscopy measurements, all of the radionuclide measurements were done with in-house procedures. The method detection limits for radiochemical measurements are dependent on both sample matrix and count time and are not listed here. In general, the

radiochemical measurements used count times to yield at least 1% (10,000 counts) counting statistics. The expected errors for the radiochemical data range from  $\pm 5$ -10 % for gross alpha/beta and gamma emitter measurements to  $\pm 10$ -20% for radionuclides that require chemical separations before counting (i.e.  $^{99}$ Tc,  $^{90}$ Sr,  $^{129}$ I, and  $^{237}$ Np).

#### 3.5 <u>Criticality Controls</u>

The current ORNL waste acceptance criteria (WAC) for liquid-low level waste requires that the fissile isotopes of uranium and plutonium be isotopically diluted with  $^{238}$ U and  $^{232}$ Th, respectively. These administrative controls require that the ratio of the  $^{238}$ U mass divided by the fissile equivalent mass (FEM) for uranium be greater than 100. The  $^{235}$ U FEM is a useful scale for criticality calculations that normalizes the fission probability for each fissile isotope to  $^{235}$ U. These FEM factors, designated as  $f_{35}$  for  $^{235}$ U mass factors, are discussed and listed in the Appendix A, Table 1 of the ORNL Procedure NCS-1.0, *Nuclear Criticality Safety Program*.

The major fissile isotopes of concern in the ORNL waste tanks are <sup>233</sup>U, <sup>235</sup>U, and <sup>239</sup>Pu. The fissile isotope <sup>241</sup>Pu is also present in the waste, but the mass is usually several orders of magnitude lower and below a level that would influence the isotopic dilution ratio for plutonium. Other fissile isotopes present in the ORNL waste include isotopes of neptunium, americium, and curium, but the actual mass present in the waste has been too low for major concern, and the low concentration would make it difficult and expensive to measure by mass spectrometry.

The data presented in this report for isotopic dilution ratios (also referred to as denature ratios) reflect both the current ORNL standard practices for disposal of fissile isotopes of uranium and plutonium. The administrative controls which were in effect when the waste was generated were different than current practices.

All calculations dealing with isotopic dilution for criticality safety are based on isotope mass ratios and must not be confused with activity ratios. For any data discussed in this report that uses <sup>232</sup>Th

relative to isotopic mass ratios, the total thorium concentration and the <sup>232</sup>Th concentration are the same value.

The current administrative requirements for criticality control are more conservative than past practices and require that the following conditions be satisfied for uranium,

$$\frac{(^{238}U) - 200(^{233}U)}{(^{235}U)} \ge 110 \tag{1}$$

$$\frac{(^{238}U) - 110(^{235}U)}{(^{233}U)} \ge 200 \tag{2}$$

The administrative controls for plutonium require a dilution ratio of 200 parts thorium to one part plutonium (past practices only required a ratio of 100).

#### 4.0 Quality Assurance

Quality assurance during the sampling activities was primarily addressed by the use of approved procedures for sampling the sludge phase found in each waste tank. These procedures provide detailed instructions for the collection, labeling, and shipping of each sample. Chain-of-custody forms were used to track individual samples from their collection point to the analytical laboratory.

The QA for the sludge characterization was based upon the RMAL Radioactive Waste Characterization QA Plan<sup>4</sup> which defines the basis for quality assurance and quality control used for the analysis of the waste tank samples. The QA plans discuss staff qualification requirements, laboratory participation in performance demonstration programs, quality control acceptance criteria for analytical methods, sample management, and most other laboratory operations. The QA plans implemented at the RMAL for waste characterization meet both the WIPP and the Nevada Test Site (NTS) QA requirements for inorganic, organic, and radiochemical measurements.

#### 5.0 Summary of Inorganic and Radiochemical Analytical Results

#### 5.1 <u>Description of Data Tables</u>

A summary of the inorganic, physical, and radiochemical analytical results for MVST sludge samples are presented in Table 2 through Table 7. Also, MVST data<sup>5</sup> collected in 1996 was included in these data tables for comparison. These tables are arranged in a similar format to facilitate comparing data from different tanks and to group information into useful units. The analytical data presented in these tables are the consolidation of data from a single project which had a fixed set of analytical requirements. Any parameter reported with a dash ("-") indicates that the data point was not collected for that sample.

The first section, "Physical properties and miscellaneous data", includes unrelated information that does not fit well into other table groups. The first parameters entered in a column include the RMAL request and sample numbers, which are laboratory filing codes used to track sample information. The next set of data includes information on the moisture or water content and the solids content of the sample. The group is completed with data on the inorganic and organic carbon content. For MVST waste tank samples the inorganic carbon can be assumed to be all carbonate and bicarbonate. The Total Organic Carbon (TOC) provides an upper limit on the organic content in the tank waste but does not include volatile organic compounds. Most of the liquid waste in the active system has been through an evaporator which removes the highly volatile organic compounds from the waste.

The next two sections include groups of metals; the "RCRA metals" are separated out for quick reference. The regulatory limit for the concentrations are listed in parentheses next to each RCRA metal. For the liquid samples, the RCRA regulatory limits are used directly, since the supernatant would be defined as the TCLP leachate in the determination of waste characteristics for hazardous waste. The RCRA metal sludge data represents total metal measurements, as defined by EPA. Exceeding the RCRA regulatory limits listed for the sludge samples only indicates that the waste has the potential to be classified as hazardous. The sludge waste should only be classified as RCRA waste if the final waste form fails the TCLP leaching test.

The remaining metals are grouped under "<u>Process metals</u>," which includes the common Group IA & IIA metals along with elements that could effect chemical processing, criticality concerns, and stabilization techniques such as grouting or vitrification. For the sludge data, all the metals are reported on a "as received" (wet weight) basis.

The section "Semi-quantitative metals by ICP-MS" includes additional metals identified in a full mass range scan by inductively-coupled plasma - mass spectrometry. This measurement helps ensure all major elements have been identified in the waste. Each element reported is not calibrated but is based upon a response factor from a curve generated from a few elements across the mass range. Therefore, these elemental concentrations are listed as estimates only.

The "Anions by ion chromatography" section includes the inorganic anions, several common water soluble organic acids. Two of the organic acids included, citrate and oxalate, are also classified as complexing agents.

The "Beta/gamma emitters" section summarizes the radionuclides that emit gamma-rays and beta particles. This section includes the gross beta activity, radionuclides identified by gamma spectrometry, and several "pure" beta emitters of interest. Many of the "pure" beta emitters (<sup>3</sup>H, <sup>14</sup>C, and <sup>90</sup>Sr) require radiochemical separations prior to measurement by either liquid scintillation or gasflow proportional counting. The <sup>99</sup>Tc was measured by ICP-MS without any prior chemical separation, and the <sup>151</sup>Sm was estimated by ICP-MS after a lanthanide group separation.

The "Alpha emitters" section summarize the actinide elements in the waste. This section includes the gross alpha activity, an estimate of the activity for each alpha emitter identified in a gross alpha spectrum, and plutonium isotopes determined by alpha spectrometry after a radiochemical separation. For supernatant samples, an estimate of the <sup>232</sup>Th/<sup>239</sup>Pu mass ratio is included in this section to address criticality concerns if enough thorium is present to calculate the ratio. For the sludge samples, this mass ratio is included with the plutonium mass spectrometry data.

The remaining sections include "<u>Uranium isotopics by ICP-MS</u>" and "<u>Plutonium isotopes by TIMS</u>." These sections summarize the uranium and plutonium data measured by mass spectrometry. Also, included in these sections are the isotopic mass dilution or "denature" ratios for uranium and plutonium based on the requirements in place when the waste was generated (see section 3.5). The plutonium section for the sludge samples also includes the activity for each plutonium isotope, which was calculated from the mass spectrometry data.

Table 2 Analytical Data for Sludge in Tanks W-24

Table 2 Analytical Data for Sludge in Tanks W-24						
Characteristic (Analysis)		1996 W-24 S	2000 W-24 S	$\mathbf{IDL}^1$		
Physical properti	es and miscella	neous data				
Request number Sample number pH		7749C 960806-006 12.8	10224 000509-001 9.8			
Water <sup>a</sup> TS <sup>b</sup> TSS <sup>c</sup> TDS <sup>d</sup> Bulk density TC <sup>e</sup> TIC <sup>f</sup> TOC <sup>g</sup>	(%) (mg/g) (mg/g) (mg/g) (g/mL) (mg/Kg) (mg/Kg) (mg/Kg)	51.2 488 - - 1.37 13700 13700 < 15	64.0 360 253 107 1.315 28800 8790 20000	- - - 15 15 15		
RCRA Metals ( ±						
Ag <sup>h</sup> (100) <sup>i</sup> As (100) Ba (2000) Cd (20) Cr (100) Hg (4) Ni (1000) Pb (100) Se (20) Tl (18)	(mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg)	< 1.9 < 5.3 75.5 13.9 61.6 38.0 45.2 303 < 5.3 < 5.3	<0.8 86.2 21.1 236 74.0 76.3 435 <0.8	0.012 0.005 0.001 0.168 0.013 0.002 0.078 0.341 0.005 0.005		
Process metals ( :	±10%)					
Al B Be Ca Co Cu Cs <sup>j</sup> Fe K Mg Mn Mo Na P Sb Si k Sr Th U V Zn	(mg/Kg)	3330 4.35 4.45 51200 2.42 28.5 0.900 1250 13400 9280 84.7 - 48800 1240 < 19 3820 283 3270 6780 2.23 479	3540 9.52 7.28 42400 22.9 67.4 0.409 2990 7980 6330 911 15.1 35200 - < 41 2340 208 7480 46500 < 1 658	0.035 0.01 0.001 0.033 0.039 0.006 0.005 0.014 0.5 0.049 0.002 0.038 0.075 0.13 0.509 0.022 0.001 0.376 0.077 0.013 0.445		

Characteristic (Analysis)		1996 W-24 S	2000 W-24 S	$\mathbf{IDL}^1$
Semi-quantitative	metals by ICP	-MS ( ±30-50 %, * indicate	es data from water leach)	
Au, gold	(mg/Kg)	1.5		-
Bi, bismuth	(mg/Kg)	170		-
Ce, cerium	(mg/Kg)	6.5		-
Er, erbium	(mg/Kg)	0.25		-
Eu, europium	(mg/Kg)	1.1		-
Ga, gallium	(mg/Kg)	5.3		-
Gd, gadolinium	(mg/Kg)	1.2		-
Ho, holmium	(mg/Kg)	1.0		-
I, iodine	(mg/Kg)	* 13		-
La, lanthanum	(mg/Kg)	9.1		-
Li, lithium	(mg/Kg)	* 170		-
Mo,molybdenum	(mg/Kg)	* 2.1		-
Nb, niobium	(mg/Kg)	0.93		-
Rb, rubidium	(mg/Kg)	* 1.4		-
Sn, tin	(mg/Kg)	12		-
Ti, titanium	(mg/Kg)	21		-
W, tungsten	(mg/Kg)	1.0		-
Zr, zirconium	(mg/Kg)	8.4		-
Anions by ion chr	omatography i	n water wash of sludge ( ±1	10%)	
Inorganic				
Bromide	(mg/Kg)	< 50	< 41	0.05
Chloride	(mg/Kg)	2770	1560	0.05
Chromate	(mg/Kg)	< 20	18.2	0.05
Fluoride	(mg/Kg)	103	468	0.05
Nitrate	(mg/Kg)	165000	67400	0.10
Nitrite	(mg/Kg)	2250	3920	0.10
Phosphate	(mg/Kg)	< 20	35.0	0.20
Sulphate	(mg/Kg)	1370	2570	0.10
<u>Organic</u>	. 2 3,			
Acetate	(mg/Kg)	242	1310	0.05
Citrate	(mg/Kg)	< 20	5.12	0.50
Formate	(mg/Kg)	175	110	0.05
Oxalate	(mg/Kg)	690	397	0.05
Phthalate	(mg/Kg)	< 20	27.9	0.05

Characteristic (Analysis)		1996 W-24 S	2000 W-24 S	$\mathbf{IDL}^1$	
Beta/gamma emi	tters ( ±10%)				
Gross beta	(Bq/g)	4.6e+06	4.9e+06	-	
<sup>59</sup> Ni	(Bq/g)	< 2.5e+01	-	-	
<sup>63</sup> Ni	(Bq/g)	3.3e+03	-	-	
<sup>60</sup> Co	(Bq/g)	2.8e+04	3.0e+04	-	
<sup>90</sup> Sr/ <sup>90</sup> Y	(Bq/g)	1.4e+06	1.3e+06	-	
<sup>99</sup> Tc	(Bq/g)	4.5e+02	-	-	
$^{129}I$	(Bq/g)	-	-	-	
<sup>134</sup> Cs	(Bq/g)	1.3e+04	-	-	
<sup>137</sup> Cs	(Bq/g)	5.3e+05	6.3e+05	-	
<sup>151</sup> Sm	(Bq/g)	< 6.0e+02	-	-	
<sup>152</sup> Eu	(Bq/g)	8.9e+04	2.4e+05	-	
<sup>154</sup> Eu	(Bq/g)	3.8e+04	2.5e+05	-	
<sup>155</sup> Eu	(Bq/g)	1.0e+04	5.1e+04	=	
<sup>227</sup> Ac	(Bq/g)	< 4.7e+03	-	=	
<sup>241</sup> Pu	(Bq/g)	1.4e+04	-	-	
Alpha emitters (	±10%)	•	•		
Gross alpha	(Bq/g)	34000	200000	_	
<sup>232</sup> Th	(Bq/g) $(Bq/g)$	13	30	_	
<sup>233</sup> U	(Bq/g) $(Bq/g)$	1600	8900	_	
<sup>234</sup> U	(Bq/g)	77	710	_	
<sup>235</sup> U	(Bq/g)	2.6	15	_	
<sup>238</sup> U	(Bq/g)	84	580	_	
<sup>237</sup> Np	(Bq/g)	10	-	_	
<sup>241</sup> Am	(Bq/g)	3900	10400	_	
<sup>244</sup> Cm	(Bq/g)	22000	155000	_	
<sup>250</sup> Cf	(Bq/g)	< 100	-	_	
<sup>252</sup> Cf	(Bq/g)	< 100	-	-	
Total Pu alpha	(Bq/g)	6600	28000	-	
<sup>238</sup> Pu	(Bq/g)	4000	17000	-	
$^{239}$ Pu/ $^{240}$ Pu	(Bq/g)	2600	11000	-	
<sup>242</sup> Pu	(Bq/g)	-	-	-	
TRU activity					
Pu+Am (3700)	(Bq/g)	10500	38400	_	
Uranium isotopics by ICP-MS (±2%)					
		· ·	1		
<sup>233</sup> U	(atom %)	0.067	0.0548	0.001	
$^{234}U$	(atom %)	0.005	0.0067	0.001	
$^{235}U$	(atom %)	0.543	0.4606	0.001	
$^{236}U$	(atom %)	0.006	0.0028	0.001	
$^{238}U$	(atom %)	99.379	99.4751	0.001	
<sup>238</sup> U/ <sup>235</sup> U FEM	-	159	189	-	

Characteristic (Analysis)		1996 W-24 S	2000 W-24 S	IDL <sup>1</sup>
Plutonium isoto	pics by TIMS ( ±	1%)		
<sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(atom %) (atom %) (atom %) (atom %) (atom %) (atom %)	0.63 87.14 10.81 0.37 1.05 < 0.01	- - - - -	- - - - -
Pu activity <sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g)	3800 1900 870 14000 1.5 < 0.1	- - - - -	- - - - -
( <sup>239</sup> Pu) <sup>232</sup> Th/ <sup>239</sup> Pu	(ng/g)	960 3920	-	- -

<sup>(</sup>a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., and (l) Instrument detection limits.

Table 3 Analytical Data for Sludge in Tanks W-25

Characteristic (Analysis)	e	1996 W-25 S	2000 W-25 S	$\mathbf{IDL}^1$
Physical prop	erties and miscellar	neous data		
Request number	er	7749D	10224	-
Sample numbe		960822-036	000509-002	-
pН		12.6	11.0	_
r				
Water <sup>a</sup>	(%)	50.9	59.5	-
$TS^b$	(mg/g)	491	406	-
TSS <sup>c</sup>	(mg/g)	-	282	-
TDS <sup>d</sup>	(mg/g)	_	124	_
Bulk density	(g/mL)	1.36	1.331	_
TC <sup>e</sup>	(mg/Kg)	15700	20900	15
TICf	(mg/Kg)	15700	11100	15
TOCg	(mg/Kg)	< 15	9800	15
100	(116/116)	\ 13	7000	10
RCRA Metals	s ( ±10%)			
Ag <sup>h</sup> (100)	i (mg/Kg)	< 1.8	_	0.012
As (100)		< 1.3	< 0.9	0.005
Ba (2000		105	73.8	0.001
Cd (20)	(mg/Kg)	11.9	26.0	0.168
Cr (100)		92.1	190	0.013
Hg (4)	(mg/Kg)	73.2	49.1	0.002
Ni (1000		56.8	82.3	0.078
Pb (100)		442	454	0.341
Se (20)	(mg/Kg)	< 1.3	< 0.9	0.005
Tl (18)	(mg/Kg)	< 1.3	< 0.9	0.005
Process metal				
		5010	2610	0.025
Al	(mg/Kg)	5810	2610	0.035
B	(mg/Kg)	3.76	7.34	0.01
Be Ca	(mg/Kg)	6.91 50800	6.40 56100	0.001 0.03
	(mg/Kg)	5.86	28.1	0.03
Co Cu	(mg/Kg)	37.0	28.1 51.4	0.039
Cu Cs <sup>j</sup>	(mg/Kg) (mg/Kg)	0.857	< 0.09	0.005
Fe	(mg/Kg)	1810	2160	0.003
re K	(mg/Kg)	8850	9820	0.014
Mg	(mg/Kg)	7650	11500	0.049
Mn		140	616	0.049
Mo	(mg/Kg) (mg/Kg)	140	37.0	0.002
Na	(mg/Kg)	52100	42000	0.038
Na P	(mg/Kg)	1850	42000	0.073
Sb	(mg/Kg)	114	< 50	0.13
Si <sup>k</sup>	(mg/Kg)	8890	2670	0.022
Sr		325	251	0.022
	(mg/Kg)	9250		0.001
Th U	(mg/Kg)	9250 7660	8960 30600	0.376
	(mg/Kg)			
V Zn	(mg/Kg) (mg/Kg)	3.85 285	< 1.2 623	0.01 0.44

Characteristic (Analysis)		1996 W-25 S	2000 W-25 S	IDL <sup>1</sup>
Semi-quantitative	metals by ICP-	MS ( ±30-50 %, * indicates	s data from water leach)	
Au, gold	(mg/Kg)	0.28		-
Bi, bismuth	(mg/Kg)	250		-
Ce, cerium	(mg/Kg)	9.4		-
Er, erbium	(mg/Kg)	0.02		-
Eu, europium	(mg/Kg)	2.1		-
Ga, gallium	(mg/Kg)	8.1		-
Gd, gadolinium	(mg/Kg)	1.7		-
Ho, holmium	(mg/Kg)	2.0		-
I, iodine	(mg/Kg)	* 12		-
La, lanthanum	(mg/Kg)	18		-
Li, lithium	(mg/Kg)	* 33		-
Mo,molybdenum	(mg/Kg)	* 2.0		-
Nb, niobium	(mg/Kg)	0.72		-
Rb, rubidium	(mg/Kg)	* 1.0		_
Sn, tin	(mg/Kg)	18		-
Ti, titanium	(mg/Kg)	47		-
W, tungsten	(mg/Kg)	0.61		-
Zr, zirconium	(mg/Kg)	16		-
Anions by ion chro	omatography ir	water wash of sludge (±1	0%)	
Inorganic				
Bromide	(mg/Kg)	< 50	< 50	0.05
Chloride	(mg/Kg)	2110	1630	0.05
Chromate	(mg/Kg)	95.5	29.5	0.05
Fluoride	(mg/Kg)	118	251	0.05
Nitrate	(mg/Kg)	162000	76600	0.10
Nitrite	(mg/Kg)	4967	3190	0.10
Phosphate	(mg/Kg)	< 20	< 10	0.20
Sulphate	(mg/Kg)	1750	2590	0.10
Organic	` <i>E E</i>			
Acetate	(mg/Kg)	318	919	0.05
Citrate	(mg/Kg)	< 20	25.5	0.50
Formate		247	130	0.05
Oxalate	(mg/Kg) (mg/Kg)	521	643	0.05
Phthalate	(mg/Kg)	< 20	18.4	0.05

Beta/gamma emitters (±10%)   Gross beta (Bq/g)   8.3e+06   4.3e+06   -	Characteristic (Analysis)		1996 W-25 S	2000 W-25 S	$\mathbf{IDL}^1$
Cross beta   (Bq'g)	(Allalysis)		W-25 S	VV-25 5	IDL
SNI	Beta/gamma emit	ters ( ±10%)			
SNI		(Bq/g)	8.3e+06	4.3e+06	-
SN	<sup>59</sup> Ni		< 2.5e+01	-	-
SCO	<sup>63</sup> Ni		3.4e+03	-	-
$ \begin{array}{c} {}^{88} F^{\mu N} Y \\ {}^{99} TC \\ {}^{129} T \\ {}^{129} T \\ {}^{129} T \\ {}^{132} GS \\ {}^{13$			2.5e+04	3.5e+04	-
PTC	<sup>90</sup> Sr/ <sup>90</sup> Y		3.2e+06	1.3e+06	-
134	<sup>99</sup> Tc		1.0e+02	-	-
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	$^{129}I$	(Bq/g)		-	-
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	<sup>134</sup> Cs	(Bq/g)	6.0e+03	_	-
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$				4.0e+05	-
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	<sup>151</sup> Sm			<del>-</del>	_
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	<sup>152</sup> Eu			6.0e+05	_
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$					_
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$					_
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$					_
Alpha emitters (±10%)   Gross alpha	<sup>241</sup> P <sub>11</sub>			_	_
Gross alpha         (Bq/g)         83000         130000         -           232Th         (Bq/g)         38         36         -           233U         (Bq/g)         2800         7200         -           234U         (Bq/g)         100         28         -           238U         (Bq/g)         3.2         7.9         -           238U         (Bq/g)         95         380         -           237Np         (Bq/g)         10         -         -           241Am         (Bq/g)         9300         10300         -           244Cm         (Bq/g)         58000         90500         -           250Cf         (Bq/g)         58000         90500         -           252Cf         (Bq/g)         13000         23000         -           252Cf         (Bq/g)         13000         23000         -           238Pu         (Bq/g)         4900         9500         -           239Pu/40Pu         (Bq/g)         -         -         -           242Pu         (Bq/g)         22300         33300         -           Uranium isotopics by ICP-MS (±2%)           233U <td></td> <td></td> <td>2.00 . 0 .</td> <td></td> <td></td>			2.00 . 0 .		
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	Alpha emitters ( ±	±10%)			
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	Gross alpha	(Bq/g)	83000	130000	-
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	<sup>232</sup> Th		38	36	-
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$			2800	7200	-
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$					-
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	<sup>235</sup> U		3.2	7.9	-
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$^{238}U$				-
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	<sup>237</sup> Np		10	_	-
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	<sup>241</sup> Am			10300	_
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	<sup>244</sup> Cm				-
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$				-	_
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$				=	-
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		\ T 0/			
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Total Pu alpha	(Bq/g)	13000	23000	-
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	<sup>238</sup> Pu				-
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	<sup>239</sup> Pu/ <sup>240</sup> Pu				-
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$			-	-	-
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	TPII activity				
Uranium isotopics by ICP-MS (±2%)       233U (atom %)     0.103 (0.0674)     0.001       234U (atom %)     0.006 (0.0004)     0.001       235U (atom %)     0.597 (0.3670)     0.001       236U (atom %)     0.006 (0.0048)     0.001	Du   Am (2700)	$(\mathbf{R}\alpha/\alpha)$	22300	33300	
233U     (atom %)     0.103     0.0674     0.001       234U     (atom %)     0.006     0.0004     0.001       235U     (atom %)     0.597     0.3670     0.001       236U     (atom %)     0.006     0.0048     0.001	1 u+AIII (3/00)	(Dq/g)	44300	33300	-
234U     (atom %)     0.006     0.0004     0.001       235U     (atom %)     0.597     0.3670     0.001       236U     (atom %)     0.006     0.0048     0.001	Uranium isotopics by ICP-MS (±2%)				
234U     (atom %)     0.006     0.0004     0.001       235U     (atom %)     0.597     0.3670     0.001       236U     (atom %)     0.006     0.0048     0.001	<sup>233</sup> U	(atom %)	0.103	0.0674	0.001
235 U     (atom %)     0.597     0.3670     0.001       236 U     (atom %)     0.006     0.0048     0.001					
<sup>236</sup> U (atom %) 0.006 0.0048 0.001					
	<sup>238</sup> U	(atom %)	99.289	99.5604	0.001
(atom /0) 97.207 97.3004 0.001		(atom 70)	77.207	77.JUU <del>4</del>	0.001
<sup>238</sup> U/ <sup>235</sup> U FEM - 137 221 -	<sup>238</sup> U/ <sup>235</sup> U FEM	-	137	221	-

Characteristic (Analysis)		1996 W-25 S	2000 W-25 S	IDL <sup>1</sup>			
Plutonium isotopics by TIMS ( ±1%)							
<sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(atom %) (atom %) (atom %) (atom %) (atom %) (atom %)	0.72 84.95 12.42 0.40 1.51 < 0.01	- - - - -	- - - - -			
Pu activity <sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g)	7800 3400 1800 26000 3.8 < 0.1	- - - - -	- - - - -			
( <sup>239</sup> Pu) <sup>232</sup> Th/ <sup>239</sup> Pu	(ng/g)	1700 6320	- -	-			

<sup>(</sup>a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., and (l) Instrument detection limits.

Table 4 Analytical Data for Sludge in Tanks W-26

Table 4 Analytical Data for Sludge in Tanks W-26								
Chara (Analy	cteristic vsis)		1996 W-26 S	2000 W-26 S	IDL¹			
Physic	Physical properties and miscellaneous data							
Reques	st number		7749E	10224	_			
			960830-044	000509-003	_			
pH	Sample number		9.7	9.1	_			
P			<i>7.,</i>	<b>7.1</b>				
Watera		(%)	50.9	56.1	_			
TS <sup>b</sup>		(mg/g)	491	439	_			
TSS <sup>c</sup>		(mg/g) (mg/g)	-	285	_			
TDS <sup>d</sup>		(mg/g) (mg/g)	_	154	_			
Bulk de	ensity	(g/mL)	1.38	1.363	_			
TC <sup>e</sup>	Chistry	(mg/Kg)	13500	10300	15			
TICf		(mg/Kg)	11600	3790	15			
TOCg		(mg/Kg)	1900	6510	15			
100		(mg/ng)	1500	0310	13			
RCRA	Metals ( ±	10%)						
$Ag^h$	$(100)^{i}$	(mg/Kg)	< 1.9	_	0.012			
As	(100)	(mg/Kg)	< 1.4	< 0.9	0.005			
Ba	(2000)	(mg/Kg)	63.1	77.9	0.001			
Cd	(20)	(mg/Kg)	19.8	21.9	0.168			
Cr	(100)	(mg/Kg)	74.4	153	0.013			
Hg	(4)	(mg/Kg)	12.7	58.3	0.002			
Ni	(1000)	(mg/Kg)	42.8	74.5	0.078			
Pb	(1000)	(mg/Kg)	212	331	0.341			
Se	(20)	(mg/Kg)	< 1.4	< 0.9	0.005			
Tl	(18)	(mg/Kg)	< 1.4	< 0.9	0.005			
Proces	Process metals (±10%)							
		·	4000	<b>-</b> 120	0.005			
Al		(mg/Kg)	1980	7130	0.035			
В		(mg/Kg)	11.3	10.6	0.01			
Be		(mg/Kg)	1.85	5.63	0.001			
Ca		(mg/Kg)	45900	43200	0.03			
Co		(mg/Kg)	2.69	21.1	0.039			
Cu		(mg/Kg)	29.0	70.6	0.006			
Cs <sup>j</sup>		(mg/Kg)	1.53	0.729	0.005			
Fe		(mg/Kg)	1010	2380	0.014 0.5			
K Ma		(mg/Kg)	25300	18400				
Mg		(mg/Kg)	14700	12800	0.049			
Mn		(mg/Kg)	102	180	0.002			
Mo		(mg/Kg)	40000	46.4	0.038			
Na		(mg/Kg)	48900	40500	0.075			
P		(mg/Kg)	1070	- - 50	0.13			
Sb S; k		(mg/Kg)	52.8	< 50	0.509			
Si k		(mg/Kg)	2100	7470	0.022			
Sr		(mg/Kg)	254	195	0.001			
Th		(mg/Kg)	3280	4330	0.376			
U		(mg/Kg)	19400	36900	0.077			
V Zn		(mg/Kg)	2.32	< 1.2 360	0.013			
ZII		(mg/Kg)	405	300	0.445			

Characteristic (Analysis)		1996 W-26 S	2000 W-26 S	$\mathbf{IDL}^1$			
Semi-quantitative metals by ICP-MS ( ±30-50 %, * indicates data from water leach)							
Au, gold	(mg/Kg)	0.92		-			
Bi, bismuth	(mg/Kg)	78		-			
Ce, cerium	(mg/Kg)	5.5		-			
Er, erbium	(mg/Kg)	0.24		-			
Eu, europium	(mg/Kg)	2.3		-			
Ga, gallium	(mg/Kg)	4.0		-			
Gd, gadolinium	(mg/Kg)	6.4		-			
Ho, holmium	(mg/Kg)	1.0		-			
I, iodine	(mg/Kg)	* 12		-			
La, lanthanum	(mg/Kg)	4.8		-			
Li, lithium	(mg/Kg)	* 76		-			
Mo,molybdenum	(mg/Kg)	* 2.2		-			
Nb, niobium	(mg/Kg)	0.22		-			
Rb, rubidium	(mg/Kg)	* 2.5		-			
Sn, tin	(mg/Kg)	7.3		-			
Ti, titanium	(mg/Kg)	3.2		-			
W, tungsten	(mg/Kg)	1.5		-			
Zr, zirconium	(mg/Kg)	5.4		-			
Anions by ion chr	omatography i	n water wash of sludge (±1	(0%)				
Inorganic							
Bromide	(mg/Kg)	< 50	< 42	0.05			
Chloride	(mg/Kg)	3070	1800	0.05			
Chromate	(mg/Kg)	< 20	4.94	0.05			
Fluoride	(mg/Kg)	< 50	119	0.05			
Nitrate	(mg/Kg)	214000	115000	0.10			
Nitrite	(mg/Kg)	1652	2210	0.10			
Phosphate	(mg/Kg)	< 20	< 8	0.20			
Sulphate	(mg/Kg)	2120	1520	0.10			
Organic	<i>\ U \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \</i>	-					
Acetate	(mg/Kg)	336	336	0.05			
Citrate	(mg/Kg)	< 20	< 4	0.50			
Formate	(mg/Kg)	243	155	0.05			
Oxalate	(mg/Kg)	44.2	32.1	0.05			
Phthalate	(mg/Kg)	< 20	< 4	0.05			

Characteristic		1996	2000			
(Analysis)		W-26 S	W-26 S	IDL <sup>1</sup>		
Beta/gamma emi	tters ( ±10%)					
Gross beta	(Bq/g)	3.5e+06	4.0e+06	-		
<sup>59</sup> Ni	(Bq/g)	< 3.0e+01	-	-		
<sup>63</sup> Ni	(Bq/g)	4.0e+03	-	-		
<sup>60</sup> Co	(Bq/g)	5.8e+04	3.1e+04	-		
<sup>90</sup> Sr/ <sup>90</sup> Y	(Bq/g)	7.1e+05	1.0e+06	-		
<sup>99</sup> Tc	(Bq/g)	1.2e+03	-	-		
$^{129}I$	(Bq/g)	-	-	-		
<sup>134</sup> Cs	(Bq/g)	1.2e+04	-	-		
<sup>137</sup> Cs	(Bq/g)	8.9e+05	8.1e+05	-		
<sup>151</sup> Sm	(Bq/g)	< 5.8e+02	-	-		
<sup>152</sup> Eu	(Bq/g)	6.4e+05	3.9e+05	-		
<sup>154</sup> Eu	(Bq/g)	2.9e+05	1.7e+05	-		
<sup>155</sup> Eu	(Bq/g)	6.3e+04	< 1.8e+04	-		
<sup>227</sup> Ac	(Bq/g)	< 9.3e+03	_	-		
<sup>241</sup> Pu	(Bq/g)	1.5e+04	-	-		
Alpha emitters ( :	±10%)	•	1			
Gross alpha	(Bq/g)	52000	48000	_		
232Th	(Bq/g) $(Bq/g)$	13	18	_		
<sup>233</sup> U	(Bq/g) $(Bq/g)$	10000	10500	_		
<sup>234</sup> U	(Bq/g) $(Bq/g)$	180	640	_		
<sup>235</sup> U	(Bq/g) $(Bq/g)$	4.0	12	_		
<sup>238</sup> U	(Bq/g) $(Bq/g)$	240	460			
<sup>237</sup> Np	(Bq/g) $(Bq/g)$	2	-	_		
<sup>241</sup> Am	(Bq/g) $(Bq/g)$	3900	4880			
<sup>244</sup> Cm	(Bq/g) $(Bq/g)$	28000	28000	_		
<sup>250</sup> Cf	(Bq/g) $(Bq/g)$	< 100	28000	_		
<sup>252</sup> Cf	(Bq/g) $(Bq/g)$	< 100	_	_		
Ci	(Dq/g)	< 100				
Total Pu alpha	(Bq/g)	7600	9100	-		
<sup>238</sup> Pu	(Bq/g)	5300	5200	-		
<sup>239</sup> Pu/ <sup>240</sup> Pu	(Bq/g)	2300	3900	-		
<sup>242</sup> Pu	(Bq/g)	-	-	-		
TOTAL AT A						
TRU activity	(D. / )	11500	14000			
Pu+Am (3700)	(Bq/g)	11500	14000	-		
Uranium isotopic	Uranium isotopics by ICP-MS ( ±2%)					
<sup>233</sup> U	(atom %)	0.152	0.0814	0.001		
$^{234}U$	(atom %)	0.004	0.0076	0.001		
$^{235}U$	(atom %)	0.296	0.4800	0.001		
$^{236}U$	(atom %)	0.006	< 0.0001	0.001		
<sup>238</sup> U	(atom %)	99.543	99.4310	0.001		
<sup>238</sup> U/ <sup>235</sup> U FEM	-	202	171	-		

Characteristic (Analysis)		1996 W-26 S	2000 W-26 S	IDL <sup>1</sup>
Plutonium isoto	opics by TIMS ( ±	1%)		
<sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(atom %) (atom %) (atom %) (atom %) (atom %) (atom %)	1.23 82.27 15.11 0.57 0.81 < 0.01	- - - - -	- - - - -
Pu activity <sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g)	5400 1300 890 15000 0.8 < 0.1	- - - - -	- - - - -
( <sup>239</sup> Pu) <sup>232</sup> Th/ <sup>239</sup> Pu	(ng/g)	700 5730	- -	- -

<sup>(</sup>a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., and (l) Instrument detection limits.

Table 5 Analytical Data for Sludge in Tanks W-27

able						
	cteristic		1996	2000	1	
(Analy	/sis)		W-27 S	W-27 S	IDL <sup>1</sup>	
Physic	Physical properties and miscellaneous data					
Reques	st number		7749F	10224	-	
	e number		960904-248	000509-004	_	
рН			12.3	10.0	-	
1						
Water		(%)	54.9	68.9	-	
$TS^b$		(mg/g)	451	311	-	
$TSS^c$		(mg/g)	-	270	-	
$TDS^d$		(mg/g)	-	41.4	-	
Bulk d	ensity	(g/mL)	1.44	1.246	-	
$TC^e$		(mg/Kg)	12400	10100	15	
$TIC^f$		(mg/Kg)	10000	< 1000	15	
TOCg		(mg/Kg)	2400	10100	15	
RCRA	Metals (±	10%)				
Ag <sup>h</sup>	(100) <sup>i</sup>	(m a/V ~)	- 10		0.012	
Ag	$(100)^{3}$ $(100)$	(mg/Kg) (mg/Kg)	< 1.8 < 1.4	- <1	0.012	
Ba	(2000)	(mg/Kg)	41.8	64.2	0.003	
Cd	(2000)	(mg/Kg)	14.8	< 16	0.168	
Cr	(100)	(mg/Kg)	55.3	132	0.013	
Hg	(4)	(mg/Kg)	29.0	196	0.002	
Ni	(1000)	(mg/Kg)	48.9	84.0	0.078	
Pb	(1000)	(mg/Kg)	157	317	0.341	
Se	(20)	(mg/Kg)	< 1.4	< 1	0.005	
Tl	(18)	(mg/Kg)	< 1.4	< 1	0.005	
Proces	ss metals ( ±	:10%)				
A 1		(m ~ /V ~)	2250	7640	0.035	
Al B		(mg/Kg) (mg/Kg)	5.98	7640 11.9	0.033	
Ве		(mg/Kg)	1.10	17.7	0.001	
Ca		(mg/Kg)	43700	26700	0.001	
Co		(mg/Kg)	2.57	16.8	0.039	
Cu		(mg/Kg)	14.2	66.3	0.006	
Cs <sup>j</sup>		(mg/Kg)	0.892	1.81	0.005	
Fe		(mg/Kg)	935	3780	0.014	
K		(mg/Kg)	6970	4880	0.5	
Mg		(mg/Kg)	7820	4800	0.049	
Mn		(mg/Kg)	65.4	287	0.002	
Mo		(mg/Kg)	-	98.5	0.038	
Na		(mg/Kg)	58200	30900	0.075	
P		(mg/Kg)	1000	-	0.13	
Sb		(mg/Kg)	37.4	< 50	0.509	
Si <sup>k</sup>		(mg/Kg)	3860	6270	0.022	
Sr		(mg/Kg)	107	163	0.001	
Th		(mg/Kg)	1290	17400	0.376	
U		(mg/Kg)	11700	29500	0.077	
V		(mg/Kg)	3.31	< 1.3	0.013	
Zn		(mg/Kg)	360	299	0.445	

Characteristic (Analysis)		1996 W-27 S	2000 W-27 S	$\mathbf{IDL^1}$
Semi-quantitative	metals by ICP	-MS ( ±30-50 %, * indicate	es data from water leach)	
Au, gold	(mg/Kg)	0.62		-
Bi, bismuth	(mg/Kg)	130		-
Ce, cerium	(mg/Kg)	7.2		-
Er, erbium	(mg/Kg)	0.12		-
Eu, europium	(mg/Kg)	0.80		-
Ga, gallium	(mg/Kg)	4.2		-
Gd, gadolinium	(mg/Kg)	1.9		-
Ho, holmium	(mg/Kg)	1.6		-
I, iodine	(mg/Kg)	* 6.8		-
La, lanthanum	(mg/Kg)	7.3		-
Li, lithium	(mg/Kg)	* 53		_
Mo,molybdenum	(mg/Kg)	* 2.0		-
Nb, niobium	(mg/Kg)	0.56		-
Rb, rubidium	(mg/Kg)	* 1.2		-
Sn, tin	(mg/Kg)	4.0		_
Ti, titanium	(mg/Kg)	99		-
W, tungsten	(mg/Kg)	1.3		-
Zr, zirconium	(mg/Kg)	4.0		1
Anions by ion chr	omatography i	n water wash of sludge (±1	10%)	
Inorganic				
Bromide	(mg/Kg)	< 50	< 41	0.05
Chloride	(mg/Kg)	2280	1190	0.05
Chromate	(mg/Kg)	< 20	16.2	0.05
Fluoride	(mg/Kg)	< 50	246	0.05
Nitrate	(mg/Kg)	210000	62500	0.10
Nitrite	(mg/Kg)	2283	2600	0.10
Phosphate	(mg/Kg)	< 20	12.1	0.20
Sulphate	(mg/Kg)	549	943	0.10
Organic	` ` ` ` ` ` ` ` ` ` ` ` ` ` ` ` ` ` ` `			
Acetate	(mg/Kg)	196	272	0.05
Citrate	(mg/Kg)	< 20	< 4.1	0.50
Formate		200	77.8	0.05
Oxalate	(mg/Kg) (mg/Kg)	16.0	228	0.05
Phthalate	(mg/Kg)	< 20	< 4.1	0.05

Characteristic		1996	2000			
(Analysis)		W-27 S	W-27 S	IDL <sup>1</sup>		
Beta/gamma emit	tters ( ±10%)					
Gross beta	(Bq/g)	1.6e+06	1.0e+07	-		
<sup>59</sup> Ni	(Bq/g)	< 2.0e+01	-	-		
<sup>63</sup> Ni	(Bq/g)	1.7e+03	_	_		
<sup>60</sup> Co	(Bq/g)	1.2e+04	9.9e+03	_		
<sup>90</sup> Sr/ <sup>90</sup> Y	(Bq/g)	4.5e+05	4.0e+06	_		
<sup>99</sup> Tc	(Bq/g)	8.7e+01	-	_		
$^{129}{ m I}$	(Bq/g)	-	<del>-</del>	_		
<sup>134</sup> Cs	(Bq/g)	< 8.1e+02	_	_		
<sup>137</sup> Cs	(Bq/g)	3.9e+05	6.0e+05	_		
<sup>151</sup> Sm	(Bq/g)	< 5.7e+02	-	_		
<sup>152</sup> Eu	(Bq/g) (Bq/g)	4.1e+04	2.2e+05			
154Eu	(Bq/g) (Bq/g)	1.7e+04	< 4.2e+04	_		
155 <b>Eu</b>		< 2.7e+03	< 4.2e+04 < 1.8e+04	- I		
<sup>227</sup> Ac	(Bq/g)	< 2.7e+03 < 6.2e+03	< 1.86+04	- I		
AC 241Pu	(Bq/g)		-	-		
Pu	(Bq/g)	6.5e+03	-	-		
Alpha emitters (	±10%)					
Gross alpha	(Bq/g)	26000	110000	-		
<sup>232</sup> Th	(Bq/g)	5.2	71	_		
<sup>233</sup> U	(Bq/g)	1000	6200	_		
<sup>234</sup> U	(Bq/g)	53	380	_		
<sup>235</sup> U	(Bq/g)	2.5	11	_		
$^{238}U$	(Bq/g)	145	360	_		
<sup>237</sup> Np	(Bq/g)	12	-	_		
<sup>241</sup> Am	(Bq/g)	2800	11600	_		
<sup>244</sup> Cm	(Bq/g) $(Bq/g)$	17000	77000	_		
<sup>250</sup> Cf	(Bq/g) $(Bq/g)$	< 100	77000	_		
<sup>252</sup> Cf	(Bq/g) (Bq/g)	< 100	_	_		
Ci	(bq/g)	< 100	-	-		
Total Pu alpha	(Bq/g)	3400	16000	_		
<sup>238</sup> Pu	(Bq/g)	2200	10000	-		
<sup>239</sup> Pu/ <sup>240</sup> Pu	(Bq/g)	1200	5800	_		
<sup>242</sup> Pu	(Bq/g)	-	-	-		
TRU activity	- ·					
Pu+Am (3700)	(Bq/g)	6200	27600	-		
Uranium isotopic	Uranium isotopics by ICP-MS ( ±2%)					
<sup>233</sup> U	(atom %)	0.025	0.0598	0.001		
<sup>234</sup> U	(atom %)	0.002	0.0057	0.001		
<sup>235</sup> U	(atom %)	0.308	0.5068	0.001		
<sup>236</sup> U	(atom %)	0.006	< 0.0001	0.001		
<sup>238</sup> U	(atom %)	99.660	99.4276	0.001		
	(atom 70)	22.UUU	77.44.7U	0.001		
<sup>238</sup> U/ <sup>235</sup> U FEM	-	296	172	-		
				l .		

Characteristic (Analysis)		1996 W-27 S	2000 W-27 S	IDL <sup>1</sup>
Plutonium isoto	opics by TIMS (±	1%)		
<sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(atom %) (atom %) (atom %) (atom %) (atom %) (atom %)	1.08 84.88 12.64 0.49 0.91 < 0.01	- - - - -	- - - - -
Pu activity <sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g)	2400 670 370 6500 0.5 < 0.1	- - - - -	- - - - -
( <sup>239</sup> Pu) <sup>232</sup> Th/ <sup>239</sup> Pu	(ng/g)	350 4390	-	- -

<sup>(</sup>a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., and (l) Instrument detection limits.

Table 6 Analytical Data for Sludge in Tanks W-28

Table 6 Analytical Data for Sludge in Tanks W-28						
Chara (Analy	cteristic ysis)		1996 W-28 S	2000 W-28 S	IDL¹	
Physic	Physical properties and miscellaneous data					
	st number e number		7749B 960724-060 12.3	10224 000509-005 11.0	- - -	
Water <sup>a</sup> TS <sup>b</sup> TSS <sup>c</sup> TDS <sup>d</sup> Bulk d TC <sup>e</sup> TIC <sup>f</sup> TOC <sup>g</sup>		(%) (mg/g) (mg/g) (mg/g) (g/mL) (mg/Kg) (mg/Kg) (mg/Kg)	47.3 527 - - 1.37 12800 10200 2600	61.7 383 266 117 1.306 8000 < 1000 8000	- - - - 15 15 15	
RCRA	Metals ( ±	10%)				
Ag <sup>h</sup> As Ba Cd Cr Hg Ni Pb Se Tl	(100) <sup>i</sup> (100) (2000) (20) (100) (4) (1000) (100) (20) (18)	(mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg)	< 1.8 < 5.0 43.3 24.9 54.8 6.55 53.6 195 < 5.0 5.97	<1 64.3 17.7 170 50.6 66.5 396 <1 <1	0.012 0.005 0.001 0.168 0.013 0.002 0.078 0.341 0.005 0.005	
Proces	ss metals ( ±	:10%)				
Al B Be Ca Co Cu Cs <sup>j</sup> Fe K Mg Mn Mo Na P Sb Si <sup>k</sup> Sr		(mg/Kg)	571 7.33 1.36 45800 3.53 28.0 0.480 599 14600 14500 91.0 - 61000 907 < 18 1080 151 1360	3860 7.14 7.62 44600 23.9 57.0 0.401 2180 8860 8760 339 28.0 37200 < 50 3000 175 4710	0.035 0.01 0.001 0.03 0.039 0.006 0.005 0.014 0.5 0.049 0.002 0.038 0.075 0.13 0.509 0.022 0.001 0.376	
U V Zn		(mg/Kg) (mg/Kg) (mg/Kg)	18500 1.54 278	31200 < 1.3 368	0.077 0.013 0.445	

Characteristic (Analysis)		1996 W-28 S	2000 W-28 S	$\mathbf{IDL}_1$		
Semi-quantitative	Semi-quantitative metals by ICP-MS ( ±30-50 %, * indicates data from water leach)					
Au, gold	(mg/Kg)	1.9		-		
Bi, bismuth	(mg/Kg)	12		-		
Ce, cerium	(mg/Kg)	7.9		-		
Er, erbium	(mg/Kg)	0.07		-		
Eu, europium	(mg/Kg)	1.5		-		
Ga, gallium	(mg/Kg)	3.1		-		
Gd, gadolinium	(mg/Kg)	6.0		-		
Ho, holmium	(mg/Kg)	0.97		-		
I, iodine	(mg/Kg)	* 9.1		-		
La, lanthanum	(mg/Kg)	2.0		_		
Li, lithium	(mg/Kg)	* 170		_		
Mo,molybdenum	(mg/Kg)	* 2.3		-		
Nb, niobium	(mg/Kg)	0.30		-		
Rb, rubidium	(mg/Kg)	* 1.9		-		
Sn, tin	(mg/Kg)	5.9		-		
Ti, titanium	(mg/Kg)	4.1		_		
W, tungsten	(mg/Kg)	1.4		-		
Zr, zirconium	(mg/Kg)	1.8		-		
Anions by ion chro	omatography i	n water wash of sludge (±1	0%)			
Inorganic						
Bromide	(mg/Kg)	< 50	< 44	0.05		
Chloride	(mg/Kg)	3460	1580	0.05		
Chromate	(mg/Kg)	< 20	10.7	0.05		
Fluoride	(mg/Kg)	< 50	70.6	0.05		
Nitrate	(mg/Kg)	248000	98800	0.10		
Nitrite	(mg/Kg)	1120	1750	0.10		
Phosphate	(mg/Kg)	< 20	< 8.8	0.20		
Sulphate	(mg/Kg)	1773	973	0.10		
Organic						
Acetate	(mg/Kg)	325	349	0.05		
Citrate	(mg/Kg)	< 20	< 4.4	0.50		
Formate	(mg/Kg) (mg/Kg)	271	90.1	0.05		
Oxalate		19.1	11.8	0.05		
Phthalate	(mg/Kg)	< 20	< 4.4	0.05		

Characteristic		1996 W-28 S	2000 W 29 S	IDL¹
(Analysis)		W-28 S	W-28 S	IDT.
Beta/gamma emit	ters ( ±10%)	<u> </u>		
Gross beta	(Bq/g)	3.1e+06	5.0e+06	-
<sup>59</sup> Ni	(Bq/g)	<2.5e+01	-	-
<sup>63</sup> Ni	(Bq/g)	3.3e+03	_	-
<sup>60</sup> Co	(Bq/g)	4.2e+04	1.5e+04	_
<sup>90</sup> Sr/ <sup>90</sup> Y	(Bq/g)	7.0e+05	1.7e+06	-
<sup>99</sup> Tc	(Bq/g)	1.2e+02	<del>-</del>	_
$^{129}I$	(Bq/g)	4.1e - 02	_	_
<sup>134</sup> Cs	(Bq/g)	< 1.2e+03	_	_
<sup>137</sup> Cs	(Bq/g)	3.1e+05	4.8e+05	_
<sup>151</sup> Sm	(Bq/g)	< 5.6e+02	-	_
<sup>152</sup> Eu	(Bq/g)	8.0e+05	5.2e+05	_
154Eu	(Bq/g) $(Bq/g)$	2.7e+05	1.4e+05	_
155Eu	(Bq/g) $(Bq/g)$	7.0e+04	< 2.0e+04	_
<sup>227</sup> Ac	(Bq/g)	< 6.7e+03	-	_
<sup>241</sup> Pu	(Bq/g) $(Bq/g)$	1.2e+04	_	_
		1.20104		
Alpha emitters ( =	±10%)			
Gross alpha	(Bq/g)	44000	66000	-
<sup>232</sup> Th	(Bq/g)	5.5	19	-
$^{233}U$	(Bq/g)	5200	4800	-
$^{234}U$	(Bq/g)	130	360	-
$^{235}U$	(Bq/g)	3.8	11	-
$^{238}U$	(Bq/g)	230	390	-
<sup>237</sup> Np	(Bq/g)	16	-	-
<sup>241</sup> Am	(Bq/g)	4600	7600	-
<sup>244</sup> Cm	(Bq/g)	25000	43000	-
<sup>250</sup> Cf	(Bq/g)	< 100	-	_
<sup>252</sup> Cf	(Bq/g)	< 100	-	-
Total Du -1-1-	(D ~/-)	4.400	11000	
Total Pu alpha <sup>238</sup> Pu	(Bq/g)	4400	11000	-
<sup>239</sup> Pu/ <sup>240</sup> Pu	(Bq/g)	2700	6400	-
<sup>239</sup> Pu/ <sup>240</sup> Pu <sup>242</sup> Pu	(Bq/g)	1700	4600	-
Pu	(Bq/g)	-	-	-
TRU activity				
Pu+Am (3700)	(Bq/g)	9000	18600	-
Uranium isotopic	s by ICP-MS (	±2%)		
<sup>233</sup> U	(atom %)	0.081	0.0441	0.001
<sup>234</sup> U	(atom %)	0.003	0.0051	0.001
<sup>235</sup> U	(atom %)	0.296	0.4827	0.001
<sup>236</sup> U	(atom %)	0.296	0.4827	0.001
<sup>238</sup> U	(atom %)	99.613	99.4647	
	(atom %)	99.013	77.404 <i>1</i>	0.001
<sup>238</sup> U/ <sup>235</sup> U FEM	-	249	186	-

Characteristic (Analysis)		1996 W-28 S	2000 W-28 S	IDL <sup>1</sup>
Plutonium isoto	pics by TIMS ( ±	1%)		
<sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(atom %) (atom %) (atom %) (atom %) (atom %) (atom %)	< 1.06 81.54 15.93 0.70 0.76 0.01	- - - - -	- - - - -
Pu activity <sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g)	3000 830 600 12000 0.5 < 0.1	- - - - -	- - - - -
( <sup>239</sup> Pu) <sup>232</sup> Th/ <sup>239</sup> Pu	(ng/g)	440 3750	- -	- -

<sup>(</sup>a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., and (l) Instrument detection limits.

Table 7 Analytical Data for Sludge in Tanks W-31

Fable 7         Analytical Data for Sludge in Tanks W-31						
	1996 W-31 S	2000 W-31 S	$\mathbf{IDL}^1$			
Physical properties and miscellaneous data						
	7749A 960717-023 9.9	10224 000509-006 9.7	- - -			
(%) (mg/g) (mg/g) (mg/g) (g/mL) (mg/Kg) (mg/Kg)	51.4 486 - 1.44 10200 5300 4900	72.5 275 196 79.2 1.210 5690 < 1000 5690	- - - - 15 15 15			
10%)						
(mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg) (mg/Kg)	< 1.9 < 5.0 124 9.03 130 70.7 104 764 < 5.0 < 5.0	<0.9 108 <15 237 65.4 70.3 717 <0.9 <0.9	0.012 0.005 0.001 0.168 0.013 0.002 0.078 0.341 0.005 0.005			
:10%)						
(mg/Kg)	12700 11.6 21.0 24100 4.76 80.2 0.543 2820 8320 2170 247 - 60600 4240 < 19 10200 174 20700 19800 7.18	7920 7.25 8.91 24700 14.1 64.3 0.395 3420 4570 2090 268 46.7 33700 - < 45 7930 88.3 8350 38600 < 1.1	0.035 0.01 0.001 0.003 0.039 0.006 0.005 0.014 0.5 0.049 0.002 0.038 0.075 0.13 0.509 0.022 0.001 0.376 0.077 0.013			
	(%) (mg/g) (mg/g) (mg/g) (mg/kg) (mg/K	1996   W-31 S	1996   W-31 S   W-31 S			

Characteristic (Analysis)		1996 W-31 S	2000 W-31 S	$\mathbf{IDL}_1$	
Semi-quantitative metals by ICP-MS ( ±30-50 %, * indicates data from water leach)					
Au, gold	(mg/Kg)	2.6		-	
Bi, bismuth	(mg/Kg)	1200		-	
Ce, cerium	(mg/Kg)	20		-	
Er, erbium	(mg/Kg)	0.85		-	
Eu, europium	(mg/Kg)	0.54		-	
Ga, gallium	(mg/Kg)	12		-	
Gd, gadolinium	(mg/Kg)	0.75		-	
Ho, holmium	(mg/Kg)	0.22		-	
I, iodine	(mg/Kg)	* 20		-	
La, lanthanum	(mg/Kg)	54		-	
Li, lithium	(mg/Kg)	* 81		-	
Mo,molybdenum	(mg/Kg)	* 1.4		-	
Nb, niobium	(mg/Kg)	2.0		-	
Rb, rubidium	(mg/Kg)	* 1.3		-	
Sn, tin	(mg/Kg)	40		-	
Ti, titanium	(mg/Kg)	34		-	
W, tungsten	(mg/Kg)	1.3		-	
Zr, zirconium	(mg/Kg)	51		-	
Anions by ion chro	matography i	n water wash of sludge (±1	0%)		
Inorganic					
Bromide	(mg/Kg)	< 50	< 50	0.05	
Chloride	(mg/Kg)	2570	817	0.05	
Chromate	(mg/Kg)	51.5	21.9	0.05	
Fluoride	(mg/Kg)	125	604	0.05	
Nitrate	(mg/Kg)	197000	51900	0.10	
Nitrite	(mg/Kg)	3470	2680	0.10	
Phosphate	(mg/Kg)	< 50	169	0.20	
Sulphate	(mg/Kg)	1090	919	0.10	
<u>Organic</u>					
Acetate	(mg/Kg)	237	< 50	0.05	
Citrate	(mg/Kg)	< 50	< 5	0.50	
Formate	(mg/Kg) (mg/Kg)	251	< 50	0.05	
Oxalate		89.8	107	0.05	
Phthalate	(mg/Kg)	< 50	< 5	0.05	

Cross beta   (Bq'g)   2.4e+07   6.4e+06   -	Characteristic		1996	2000		
Gross beta         (Bq/g)         2.4e+07         6.4e+06         - <sup>39</sup> Ni         (Bq/g)         < 3.3e+01         -         - <sup>40</sup> Ni         (Bq/g)         4.4e+03         -         - <sup>40</sup> Co         (Bq/g)         2.2e+04         < 6.4e+03         - <sup>40</sup> Sr. <sup>40</sup> Y         (Bq/g)         1.1e+07         2.6e+06         - <sup>40</sup> TC         (Bq/g)         1.1e+02         -         - <sup>134</sup> CS         (Bq/g)         1.4e+02         -         - <sup>134</sup> CS         (Bq/g)         2.5e+03         -         - <sup>135</sup> CS         (Bq/g)         2.5e+03         -         - <sup>135</sup> CS         (Bq/g)         4.3e+05         5.7e+05         - <sup>135</sup> Sm         (Bq/g)         3.0e+04         < 3.0e+04         - <sup>135</sup> Eu         (Bq/g)         2.0e+04         < 1.9e+04         - <sup>135</sup> Eu         (Bq/g)         2.34e+03         < 1.1e+04         - <sup>235</sup> Lu         (Bq/g)         2.5e+03         -         - <sup>347</sup> Pu         (Bq/g)         3.4e+03         -         -	(Analysis)		W-31 S	W-31 S	IDL <sup>1</sup>	
Section   Sec	Beta/gamma emit	iters ( ±10%)				
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	Gross beta	(Bq/g)	2.4e+07	6.4e+06	-	
αNi         (Bq'g)         4.4e+03         -           αCo         (Bq'g)         2.2e+04         < 6.4e+03         -           αSp. ανγ         (Bq'g)         1.1e+07         2.6e+06         -           στε         (Bq'g)         1.4e+02         -         -           12° [1]         (Bq'g)         4.5e+02         -         -           13°Cs         (Bq'g)         4.3e+05         5.7e+05         -           13°Sta         (Bq'g)         3.0e+04         < 3.0e+04         -           15°Eu         (Bq'g)         3.0e+04         < 1.9e+04         -           15°Eu         (Bq'g)         2.0e+04         < 1.9e+04         -           15°Eu         (Bq'g)         2.3e+03         -         -           22°Ac         (Bq'g)         < 3.4e+03         < 1.1e+04         -           15°Eu         (Bq'g)         < 2.5e+03         -         -           22°Ac         (Bq'g)         < 2.4e+04         -         -           22°Ac         (Bq'g)         < 2.4e+04         -         -           22°Th         (Bq'g)         310         860         -           22°Th         (Bq'g)         1	<sup>59</sup> Ni		< 3.3e+01	_	_	
69Co         (Bq/g)         2.2e+04         < 6.4e+03         -           98Sr/97Y         (Bq/g)         1.1e+07         2.6e+06         -           97Tc         (Bq/g)         1.1e+07         2.6e+06         -           13°I         (Bq/g)         1.4e+02         -         -           13°ICS         (Bq/g)         4.5e+02         -         -           13°ISTA         (Bq/g)         4.3e+05         5.7e+05         -           13°ISTA         (Bq/g)         3.0e+04         < 3.0e+04         -           13°ISTA         (Bq/g)         3.0e+04         < 3.0e+04         -           13°ISTA         (Bq/g)         2.0e+04         < 1.9e+04         -           13°ISTA         (Bq/g)         3.4e+03         < 1.1e+04         -           23°ID         (Bq/g)         < 5.8e+03         < 1.1e+04         -           22°ID         (Bq/g)         < 5.8e+03         < 1.1e+04         -           22°ID         (Bq/g)         84         34         -           22°ID         (Bq/g)         84         34         -           22°ID         (Bq/g)         5200         1970         -           22°ID<	<sup>63</sup> Ni			_	_	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				< 6.4e+03	_	
9°TC (Bq'g) 1.4e+02					_	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$					_	
134Cs				_	_	
$\begin{array}{c} ^{137}{\rm Cs} & (Bq'g) & 4.3 \text{e} + 0.5 \\ ^{129}{\rm Sm} & (Bq'g) & < 6.0 \text{e} + 0.2 \\ ^{129}{\rm Em} & (Bq'g) & 3.0 \text{e} + 0.4 \\ ^{129}{\rm Eu} & (Bq'g) & 3.0 \text{e} + 0.4 \\ ^{129}{\rm Eu} & (Bq'g) & 2.0 \text{e} + 0.4 \\ ^{129}{\rm Eu} & (Bq'g) & 3.3 \text{e} + 0.4 \\ ^{129}{\rm Eu} & (Bq'g) & 3.3 \text{e} + 0.4 \\ ^{129}{\rm Eu} & (Bq'g) & 3.4 \text{e} + 0.3 \\ ^{221}{\rm Pu} & (Bq'g) & 2.4 \text{e} + 0.4 \\ \end{array}$ $\begin{array}{c} - & - & - & - \\ - & - & - & - \\ - & - &$				_	_	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$				5.7e+05	_	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$				5.76103	_	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$				< 3 Oa + O4	_	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$					-	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$					-	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$					-	
Alpha emitters (±10%)         Gross alpha (Bq/g)       160000       43000       -         223Th (Bq/g)       84       34       -         233U (Bq/g)       5200       1970       -         234U (Bq/g)       310       860       -         235U (Bq/g)       10       17       -         238U (Bq/g)       244       480       -         237Np (Bq/g)       21       -       -         241Am (Bq/g)       14000       2900       -         244Cm (Bq/g)       110000       29000       -         230Cf (Bq/g)        -       -         250Cf (Bq/g)        -       -         252Cf (Bq/g)        -       -         252Cf (Bq/g)        -       -         252Pu (Bq/g)        -       -         238Pu (Bq/g)       6200       4100       -         238Pu (Bq/g)       6200       4100       -         239Pu/240Pu (Bq/g)       6200       4100       -         242Pu (Bq/g)       -       -       -         Vu+Am (3700)       Bq/g)       33000       12200       - <td co<="" td=""><td>241D</td><td></td><td></td><td><del>-</del></td><td>-</td></td>	<td>241D</td> <td></td> <td></td> <td><del>-</del></td> <td>-</td>	241D			<del>-</del>	-
Gross alpha         (Bq/g)         160000         43000         -           232Th         (Bq/g)         84         34         -           233U         (Bq/g)         5200         1970         -           234U         (Bq/g)         310         860         -           235U         (Bq/g)         10         17         -           238U         (Bq/g)         244         480         -           237Np         (Bq/g)         21         -         -           244Am         (Bq/g)         14000         2900         -           244Cm         (Bq/g)         110000         29000         -           250Cf         (Bq/g)         <100	Pu	(Bq/g)	2.46+04	-	-	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Alpha emitters (	±10%)				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Gross alpha	(Ba/g)	160000	43000	-	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$					_	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$					_	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	234 <b>[</b> ]				_	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$					_	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$					_	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$				-	_	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	241 A m			2000		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	244Cm				-	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	250Cf			29000	-	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$				-	-	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	CI	( <b>b</b> q/g)	< 100	-	=	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Total Pu alpha	(Bq/g)	19000	9300	-	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	<sup>238</sup> Pu		13000	5200	-	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$^{239}$ Pu/ $^{240}$ Pu		6200	4100	-	
Pu+Am (3700)       (Bq/g)       33000       12200       -         Uranium isotopics by ICP-MS ( $\pm 2\%$ )         233U       (atom %)       0.075       0.0146       0.001         234U       (atom %)       0.007       0.0098       0.001         235U       (atom %)       0.750       0.6333       0.001         236U       (atom %)       0.004       < 0.0001	<sup>242</sup> Pu		-	-	-	
Pu+Am (3700)       (Bq/g)       33000       12200       -         Uranium isotopics by ICP-MS ( $\pm 2\%$ )         233U       (atom %)       0.075       0.0146       0.001         234U       (atom %)       0.007       0.0098       0.001         235U       (atom %)       0.750       0.6333       0.001         236U       (atom %)       0.004       < 0.0001	TDII aativitee					
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$		(Da/~)	22000	12200		
233 U     (atom %)     0.075     0.0146     0.001       234 U     (atom %)     0.007     0.0098     0.001       235 U     (atom %)     0.750     0.6333     0.001       236 U     (atom %)     0.004     < 0.0001	Pu+Am (3/00)	(Bq/g)	33000	12200	-	
234U     (atom %)     0.007     0.0098     0.001       235U     (atom %)     0.750     0.6333     0.001       236U     (atom %)     0.004     < 0.0001	Uranium isotopic	s by ICP-MS (	±2%)			
234U     (atom %)     0.007     0.0098     0.001       235U     (atom %)     0.750     0.6333     0.001       236U     (atom %)     0.004     < 0.0001	$^{233}U$	(atom %)	0.075	0.0146	0.001	
235 U       (atom %)       0.750       0.6333       0.001         236 U       (atom %)       0.004       < 0.0001						
236U (atom %) 0.004 < 0.0001 0.001 238U (atom %) 99.165 99.3423 0.001						
<sup>238</sup> U (atom %) 99.165 99.3423 0.001						
<sup>238</sup> U/ <sup>235</sup> U FEM - 118 154 -		(410111 /0)	77.103	77.3 123	0.001	
ı	<sup>238</sup> U/ <sup>235</sup> U FEM	-	118	154	-	

Characteristic (Analysis)		1996 W-31 S	2000 W-31 S	IDL <sup>1</sup>
Plutonium isoto	opics by TIMS ( ±	1%)		
<sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(atom %) (atom %) (atom %) (atom %) (atom %) (atom %)	< 1.16 81.94 14.55 0.34 1.9 0.11	- - - - -	- - - - -
Pu activity <sup>238</sup> Pu <sup>239</sup> Pu <sup>240</sup> Pu <sup>241</sup> Pu <sup>242</sup> Pu <sup>244</sup> Pu	(Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g) (Bq/g)	13000 3400 2200 24000 5.1 < 0.1	- - - - -	- - - - -
( <sup>239</sup> Pu) <sup>232</sup> Th/ <sup>239</sup> Pu	(ng/g)	1820 13800	- -	- -

<sup>(</sup>a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., and (l) Instrument detection limits.

#### 5.2 <u>Discussion of MVST Sludge Characteristics</u>

Determination of the mass and charge balance for a sludge sample typically has a larger error bar than what is observed with a supernatant sample. The assumptions required about the chemical form and the oxidation state of the species present in the sludge are not well known. Many of the compounds in the sludge are mixed oxides and can not be directly measured. The sludge is actually a slurry with a high water content. The interstitial liquid is in close contact with the sludge, and there are many ionic solubility equilibriums. The anion data for the sludge samples discussed in this report are based on the water soluble anions that would be available to a water wash. The water wash does not account for the insoluble hydroxides, carbonates, and mixed oxides present in a sludge sample. The insoluble species do not contribute to the charge balance, and the cation charge is not used in the calculation, as indicated in Table 8. Most of the nitrate reported for the sludge is due to the interstitial liquid. Considering these limitations, the compounds listed in Table 8 were used to estimate the mass and charge balance.

Table 8 Assumption Used for Major Compounds in MVST Sludge

Cation	Chemical Form	Cation Charge Used	Gravimetric Factors
$Al^{3+}$	$Al_2O_3$	0	1.890
Ca <sup>2+</sup>	CaCO <sub>3</sub>	0	2.497
$\mathrm{Fe^{3+}}$	$Fe_2O_3$	0	1.430
$\mathbf{K}^{+}$	$K^+NO_3^-$	+1	2.586
$\mathrm{Mg}^{2+}$	$Mg(OH)_2$	0	2.399
Mn <sup>2+</sup>	$Mn(OH)_2$	0	1.619
Na <sup>+</sup>	Na <sup>+</sup> NO <sub>3</sub> <sup>-</sup>	+1	3.697
$\mathrm{Th}^{4+}$	$Th(OH)_4$	0	1.293
$\mathrm{UO_2}^{2+}$	$UO_2((OH)_2-H_2O$	0	1.353

Table 9 summarizes the mass and charge balance for the MVST tank sludge samples. Considering the limitations of these calculations, the mass balance is within the analytical error ( $\pm 20\%$ ) for these sludge samples. The charge balance is more influenced by the chemical form assumptions, and the results have a larger corresponding error range.

Table 9 Summary of Quality Checks for MVST Sludge Data

Tank	Mass Balance (TS <sub>calc.</sub> /TS <sub>meas.</sub> ) <sup>a</sup>	Charge Balance (M <sup>+</sup> /A <sup>-</sup> )	pН	<sup>134</sup> Cs+ <sup>137</sup> Cs (%)	<sup>90</sup> Sr/ <sup>90</sup> Y (%)	Beta Recovery (%)
W-24	1.01	1.09	9.8	18.3	64.6	85.7
W-25	1.03	1.16	11.0	12.9	71.9	86.8
W-26	0.97	1.05	9.1	26.8	56.7	89.3
W-27	0.97	1.30	10.0	7.9	90.0	89.9
W-28	0.94	1.09	11.0	12.2	74.2	92.9
W-31	1.10	1.62	9.7	11.3	88.6	92.7

<sup>&</sup>lt;sup>a</sup> Mass balance is based on the calculated verses measured total solids (TS).

The beta recovery results are listed in Table 9. As discussed before, the variability for the beta recovery is probably due to the analytical error on the <sup>90</sup>Sr measurement. Any measurement error for the <sup>90</sup>Sr activity would be doubled when considering the beta recovery calculation.

The distribution, by weight percent, of the major compounds from Table 8 are illustrated in Fig. 1 for each MVST sludge sample. The distribution of the total uranium and thorium concentrations in the MVST sludge samples are shown in Fig. 2, and the change in uranium concentration between sludge sample collected in 1996 and 2000 is shown in Fig. 3.

Figure 1 Distribution of Major Compounds in the MVST Sludge

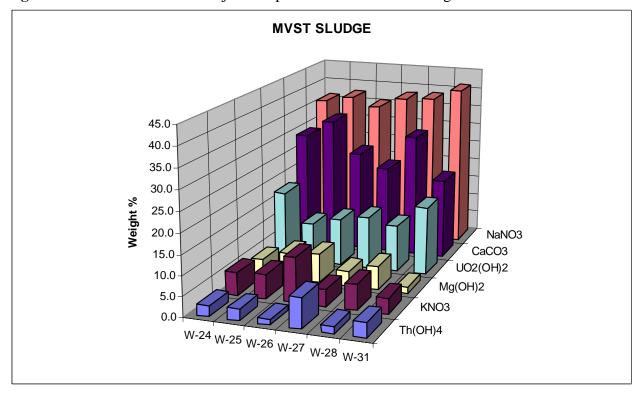


Figure 2 Distribution of Uranium and Thorium in the MVST Sludge

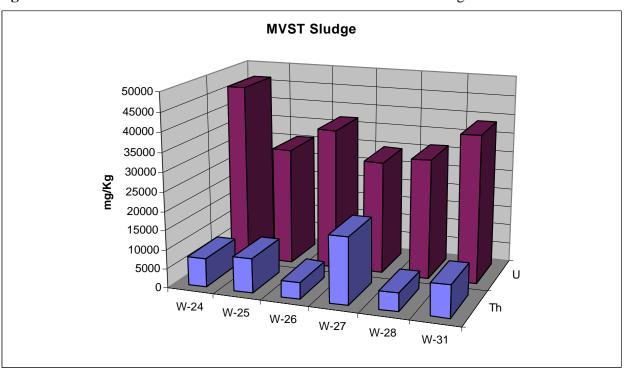
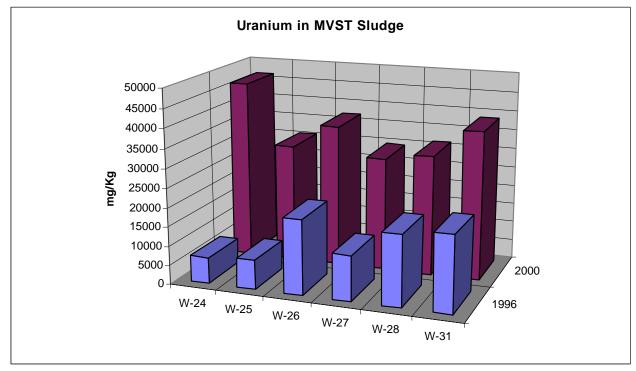


Figure 3 Distribution of Uranium in the MVST Sludge in 1996 and 2000



The distribution of the major beta emitters found in the MVST sludge samples are summarized in Table 10. The distributions of the beta activity are shown to be dependent upon the radionuclides present, which is a function of the age of the radioactive waste, and the pH of the supernatant found over the sludge. Under the typical basic conditions for ORNL waste tanks, the major difference in the beta distribution between the supernatant and the sludge is that the distribution of the longer lived fission products ( $^{90}$ Sr and  $^{137}$ Cs) are reversed due to the differences in solubility. The Group IA metals ( $^{134}$ Cs and  $^{137}$ Cs) and the radionuclides that form anionic species ( $^{99}$ TcO<sub>4</sub>,  $^{129}$ Ir, and  $^{129}$ IO<sub>3</sub>) are more soluble in the supernatant. The solubility of the Group IIA metals ( $^{90}$ Sr) in the supernatant are a function of both pH and carbonate concentration. At high pH, most of the other metals, lanthanides, and actinide elements form insoluble hydroxides and mixed oxides, which are found in the sludge. The  $^{99}$ Tc activity is higher in the supernatant than the sludge. The source of most of the  $^{99}$ Tc found in the sludge samples was the interstitial liquid, and not insoluble forms of technetium. The shorter lived radionuclides observed include the europium ( $^{152}$ Eu,  $^{154}$ Eu, and  $^{155}$ Eu) isotopes and to some extent  $^{134}$ Cs.

 Table 10
 Distribution of Beta Activity in the MVST Sludge

		Percent of Total Beta Activity								
Tank pH	<sup>90</sup> Sr/ <sup>90</sup> Y (%)	<sup>134</sup> Cs+ <sup>137</sup> Cs (%)	<sup>60</sup> Co (%)	<sup>99</sup> Tc (%)	<sup>152,154,155</sup> Eu (%)	<sup>241</sup> Pu (%)				
W-24	9.8	64.6	18.3	0.8	< 0.1	16.1	< 0.1			
W-25	11.0	71.8	12.9	1.0	< 0.1	13.9	< 0.1			
W-26	9.1	56.7	26.8	0.9	< 0.1	15.5	< 0.1			
W-27	10.0	90.0	7.9	0.1	< 0.1	1.9	< 0.1			
W-28	11.0	74.2	12.2	0.3	< 0.1	13.1	< 0.1			
W-31	9.7	88.6	11.3	< 0.1	< 0.1	< 0.1	< 0.1			

Table 11 Summary of Actinide Elements in MVST Sludge

	W-24	W-25	W-26	W-27	W-28	W-31
Actinide	(% á)					
<sup>232</sup> Th	0.04	0.05	0.03	0.02	0.01	0.06
$^{233}{ m U}$	4.68	3.36	20.12	4.11	13.20	3.51
$^{234}{ m U}$	0.23	0.12	0.36	0.22	0.33	0.21
$^{235}{ m U}$	< 0.01	< 0.01	< 0.01	0.01	0.01	< 0.01
$^{238}{ m U}$	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
<sup>237</sup> Np	0.03	0.01	< 0.01	0.05	0.04	0.01
<sup>238</sup> Pu	11.12	9.37	10.87	9.87	7.62	8.77
<sup>239</sup> Pu	5.56	4.08	2.62	2.76	2.11	2.29
<sup>240</sup> Pu	2.55	2.16	1.79	1.52	1.52	1.48
$^{241}Am^a$	11.41	11.17	7.85	11.52	11.68	9.45
<sup>244</sup> Cm	64.38	69.67	56.35	69.92	63.47	74.21
Gross á (Bq/g)	34000	83000	52000	26000	44000	160000

 $<sup>^</sup>a$  The  $^{241}Am$  data is based on subtracting the  $^{238}Pu$  by ICP-MS from the alpha peak measured at 5.15 MeV ( $^{238}Pu+^{241}Am$ ) in the alpha spectrum.

The distribution of the alpha activity is summarized in Table 11, which includes the percent alpha for each MVST sludge sample. In general, the alpha activity in the MVST system is strongly weighted by the <sup>244</sup>Cm, which has a high specific activity. The list of actinides in Table 11 required several radiochemical and inorganic analytical measurements to generate the best estimates for each of the alpha activities. The <sup>232</sup>Th activity is calculated from the total thorium measured by ICP-AES. The other thorium isotopes (<sup>228</sup>Th, <sup>229</sup>Th, and <sup>230</sup>Th) are present in the ORNL sludge waste at such low mass, their presence would not effect the ICP-AES measurement. The uranium isotopes are measured by ICP-MS. The atom % results are converted to weight %, which is used to calculate the concentration of each uranium isotope from the total uranium results obtained by ICP-AES. The activity for each uranium radionuclide is then calculated from the specific activity for each isotope. The plutonium isotopes are first measured by ICP-MS, and the total plutonium alpha activity, measured after a chemical separation, is used to calculate the activity for each isotope. The <sup>244</sup>Cm was measured directly by alpha spectrometry without any chemical separation. The <sup>241</sup>Am activity is determined by subtracting the <sup>238</sup>Pu activity from the sum of the <sup>238</sup>Pu + <sup>241</sup>Am measured by alpha spectrometry. Both <sup>238</sup>Pu and <sup>241</sup>Am have an alpha energy of about 5.50 MeV and can not be resolved by alpha spectrometry. There was no chemical separation of the plutonium and americium for this project because of cost concerns.

# 5.4 RCRA Characteristics for the MVST System

The RCRA regulatory limits are listed in Table 12, which also includes the limits for the EPA Toxicity Characteristic Leaching Protocol (TCLP) extract and the functional total metal limits for a solid or sludge waste. The total metal limits are a factor of twenty times higher than the TCLP extraction limits and are based on the 1:20 dilution used for the TCLP extraction procedure.

Table 12 Summary of RCRA Regulatory Limits

Metals		TCLP Extract and Liquids (mg/L)	Solid/Sludge Total Metal (mg/Kg)
Silver	(Ag)	5	100
Arsenic	(As)	5	100
Barium	(Ba)	100	2000
Cadmium	(Cd)	1	20
Chromium	(Cr)	5	100
Mercury	(Hg)	0.2	4
Nickel	(Ni)	50	1000
Lead	(Pb)	5	100
Selenium	(Se)	1	20
Thallium	(Tl)	0.9	18

If the RCRA metal concentrations are found to be below the total metal limits, the solid waste can not fail the TCLP leach test. If the RCRA metal concentrations exceed the total metal limits, the TCLP leach test must be done to determine if the solid waste is hazardous. For solid samples, the TCLP leach test is only valid for the final waste form ready for disposal. The total metal concentration data can be used as acceptable process knowledge if the final waste form only results in a dilution of the RCRA metal concentrations. Examples of waste forms that result in a dilution of a solid waste includes grouting (2 fold dilution) and vitrification (3 fold dilution). If the total metal limit is exceeded after stabilizing the waste, the TCLP leach test would be required for only the metals that had the potential to exceed the regulatory limits.

All of the MVST tank sludge samples exceed the total metal limits for lead and mercury, and two tanks are over or near the limit for chromium. Most of the ORNL radioactive waste sludge samples, characterized to date, have exceeded the total metal limits for these three RCRA metals. Based on past experience, it is expected that solidification of the ORNL MVST sludge would fix these RCRA metals such that the final waste form would pass the TCLP leach test.

Table 13 Summary of the MVST Sludge TCLP Data

MVST	Tanks		Sludge	Water Wash <sup>a</sup>	TCLP Extract
W-24	Cd	(mg/L)	21.1	0.975	0.476
	Cr	(mg/L)	236	2.64	0.144
	Hg	(mg/L)	74	0.302	0.177
	Pb	(mg/L)	435	0.390	0.0434
	Gross α	(Bq/mL)	200000	180	-
	$^{232}\text{U}+^{228}\text{Th}$	(Bq/mL)	-	9.7	-
	$^{233}U+^{234}U$	(Bq/mL)	9600	78	-
	$^{238}U$	(Bq/mL)	580	5.2	-
	$^{239}$ Pu+ $^{240}$ Pu	(Bq/mL)	11000	< 0.1	-
	$^{238}$ Pu+ $^{241}$ Am	(Bq/mL)	27000	8.6	-
	<sup>244</sup> Cm	(Bq/mL)	155000	79	-
W-25	Cd	(mg/L)	26.0	1.11	0.409
	Cr	(mg/L)	190	4.5	0.327
	Hg	(mg/L)	49.1	0.22	0.127
	Pb	(mg/L)	454	0.508	0.0391
	Gross α	(Bq/mL)	130000	70	-
	$^{232}U+^{228}Th$	(Bq/mL)	-	< 0.1	-
	$^{233}U + ^{234}U$	(Bq/mL)	7200	2.6	-
	$^{238}U$	(Bq/mL)	380	0.21	-
	$^{239}$ Pu+ $^{240}$ Pu	(Bq/mL)	9200	3.4	-
	$^{238}$ Pu+ $^{241}$ Am	(Bq/mL)	24000	12	-
	<sup>244</sup> Cm	(Bq/mL)	90500	51	-
W-26	Cd	(mg/L)	21.9	< 0.84	0.399
	Cr	(mg/L)	153	1.28	0.0752
	Hg	(mg/L)	58.3	2.75	0.503
	Pb	(mg/L)	331	0.353	0.0401
	Gross α	(Bq/mL)	48000	29	-
	$^{232}U+^{228}Th$	(Bq/mL)	-	< 0.1	-
	$^{233}U+^{234}U$	(Bq/mL)	11000	6.2	-
	$^{238}U$	(Bq/mL)	460	0.93	-
	$^{239}$ Pu+ $^{240}$ Pu	(Bq/mL)	3900	1.9	-
	$^{238}$ Pu+ $^{241}$ Am	(Bq/mL)	10000	5.2	-
	<sup>244</sup> Cm	(Bq/mL)	28000	15	-

MVST	Tanks		Sludge	Water Wash <sup>a</sup>	TCLP Extract
W-27	Cd	(mg/L)	< 16	< 0.84	< 0.28
	Cr	(mg/L)	132	2.50	0.207
	Hg	(mg/L)	196	2.09	0.626
	Pb	(mg/L)	317	0.141	0.0361
	Gross α	(Bq/mL)	110000	32	-
	$^{232}U+^{228}Th$	(Bq/mL)	-	< 0.1	-
	$^{233}U + ^{234}U$	(Bq/mL)	6600	1.3	-
	$^{238}U$	(Bq/mL)	360	0.16	-
	$^{239}$ Pu+ $^{240}$ Pu	(Bq/mL)	5800	1.7	-
	$^{238}$ Pu $+^{241}$ Am	(Bq/mL)	22000	6.3	-
	<sup>244</sup> Cm	(Bq/mL)	77000	23	-
W-28	Cd	(mg/L)	17.7	< 0.84	0.384
	Cr	(mg/L)	170	1.95	0.279
	Hg	(mg/L)	50.6	0.307	0.150
	Pb	(mg/L)	396	0.585	< 0.08
	Gross α	(Bq/mL)	66000	52	-
	$^{232}U+^{228}Th$	(Bq/mL)	-	< 0.1	-
	$^{233}U+^{234}U$	(Bq/mL)	5200	3.8	-
	$^{238}U$	(Bq/mL)	390	0.31	-
	$^{239}$ Pu+ $^{240}$ Pu	(Bq/mL)	3800	3.8	-
	$^{238}$ Pu+ $^{241}$ Am	(Bq/mL)	14000	11	-
	<sup>244</sup> Cm	(Bq/mL)	43000	33	-
W-31	Cd	(mg/L)	< 15	< 0.84	< 0.28
	Cr	(mg/L)	237	2.96	0.352
	Hg	(mg/L)	65.4	0.448	0.195
	Pb	(mg/L)	717	0.096	0.0805
	Gross α	(Bq/mL)	43000	24	-
	$^{232}U+^{228}Th$	(Bq/mL)	-	1.5	-
	$^{233}U + ^{234}U$	(Bq/mL)	2800	15	-
	$^{238}U$	(Bq/mL)	480	4.1	-
	$^{239}$ Pu+ $^{240}$ Pu	(Bq/mL)	4000	< 0.1	-
	$^{238}$ Pu+ $^{241}$ Am	(Bq/mL)	8100	< 0.1	-
	<sup>244</sup> Cm	(Bq/mL)	29000	3.0	-

<sup>&</sup>lt;sup>a</sup> Liquid phase from 5:1 water wash of sludge.

#### 5.5 TRU Classifications for LLLW System

The DOE definition for Transuranic (TRU) Waste includes the following conditions,

- ! TRU activity  $\geq 3700 \text{ Bq/g} (100 \text{ nCi/g}),$
- ! TRU isotopes must be alpha emitting actinide(s) with Z > 92 (uranium),
- ! TRU isotopes must have a half life  $\geq 20$  years.

This definition excludes all thorium and uranium isotopes. The short lived actinide  $^{244}$ Cm ( $t_{1/2}$  = 18.1 years), which is common to ORNL waste, falls outside the TRU definition. Also, the plutonium isotope,  $^{241}$ Pu, would be excluded from calculation of the TRU activity because it is a pure beta emitter. The primary actinide elements common to ORNL waste, that are present at sufficient levels to meet the TRU definition, include  $^{238}$ Pu,  $^{239}$ Pu,  $^{240}$ Pu, and  $^{241}$ Am. There is some current work at the Radiochemical Engineering Development Center (Mark-42 fuel assembly processing) that could generate enough  $^{243}$ Am to make a significant contribution to TRU alpha content of the waste. The remaining actinide elements present in ORNL waste are generally not available at high enough activity, and/or do not have a long enough half-life to meet the TRU definition.

None of the MVST supernatant samples discussed in this report had enough alpha activity to be considered as TRU waste. All of the MVST sludge that has been characterized to date has been classified as TRU waste based on only the plutonium and americium activity. The alpha activity reported is based on wet weight, if adjusted for dry weight the activity would almost double. The MVST sludge samples contained enough plutonium and americium activity to easily satisfy the WIPP waste acceptance criteria<sup>12</sup> for transuranic waste. Based on the TRU activity, any dilution of the sludge that would result from a solidification process such as grouting or vitrification would most likely not effect the TRU classification.

#### 5.6 <u>Distribution of Fissile Material in LLLW System</u>

As discussed in section 3.5, the ORNL LLLW waste acceptance criteria (WAC) requires the fissile isotopes of uranium and plutonium to be diluted with <sup>238</sup>U and <sup>232</sup>Th, respectively. A summary of the dilution ratios for fissile material in the sludge samples is provided in Table 14. All the dilution ratios for the MVST sludge samples exceed the required dilution factors for the fissile isotopes of uranium

and plutonium. All the dilution ratios listed in Table 14 are based on equations discussed in section 3.5 of this report.

Table 14 Summary of Denature Ratios for MVST Sludge

Tank	$^{238}\mathrm{U}/^{235}\mathrm{U}f_{35}$ (eq. 1)	<sup>238</sup> U/ <sup>233</sup> U (eq. 2)	рН
W-24	195	922	12.8
W-25	238	905	12.6
W-26	176	594	9.7
W-27	175	758	12.3
W-28	191	1090	12.3
W-31	154	2138	9.9

The dilution ratios listed in Tables 14 and 15 are based on the ratio of weight %, not the ratio of atom % given in the data tables. There is a small difference between atom %, reported for the uranium and plutonium, and weight %, which is needed for many calculations performed with the analytical data. To convert from atom % to weight %, we used the following equation,

$$W_i = \frac{a_i M_i}{\sum_{i=1}^{n} a_i M_i} X 100$$

where, 
$$\begin{aligned} W_i &= weight \,\%, \\ M_i &= nuclidic \; mass \\ a_i &= atom \,\%. \end{aligned}$$

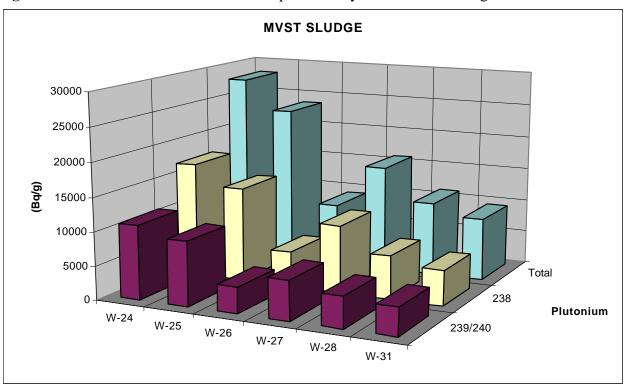
An example of this calculation is provided in Table 15, which shows there is not much difference between the atom % and the weight %.

Table 15 Example of Converting Atom % to Weight % for W-31 Sludge

Isotope	Nuclidic mass (g/mol)	atom %	$(\mathbf{a}_{\mathrm{i}}\mathbf{M}_{\mathrm{i}})$	weight %
<sup>233</sup> U	233.039629	0.056	13.0502	0.0548
$^{234}U$	234.040947	0.004	0.9362	0.0039
$^{235}U$	235.043924	0.621	145.9623	0.6132
$^{236}$ U	236.045563	0.002	0.4721	0.0020
$^{238}U$	238.050785	99.316	23642.2518	99.3260
Total		99.999	23802.6726	99.9999

The distribution of plutonium isotopes by alpha activity are illustrated in Fig. 5 for each of the MVST samples. One should note that the  $^{238}$ Pu dominates the alpha activity and the  $^{239}$ Pu is the major isotope by weight or concentration.

Figure 4 Distribution of Plutonium Alpha Activity in the MVST Sludge



### 5.7 <u>Estimates for Compliance with WIPP WAC, Rev. 5 for MVST Sludge</u>

The purpose of this section is to establish upper boundary estimates, based upon a 55-gal. drum shipping container, for several of the nuclear properties criteria and requirements for RH-TRU waste as specified in the WIPP WAC, Revision 5. Specifically, this section will develop estimates for the <sup>239</sup>Pu Fissile Gram Equivalent (FGE), <sup>239</sup>Pu Equivalent Activity, and Thermal Power or decay heat limits per RH-TRU canister. The RH-TRU limits per waste canister for each of these nuclear criteria are listed as follows,

! 239Pu FGE < 325 g ! 239Pu Equivalent Activity < 1000 Ci ! Thermal Power < 300 watts.

For the MVST sludge, the <sup>239</sup>Pu FGE can be estimated by the summation of the gram-equivalents for <sup>233</sup>U, <sup>235</sup>U, and <sup>239</sup>Pu. As shown in Table 16, the <sup>235</sup>U dominates the total <sup>239</sup>Pu FGE for the MVST sludge samples and the <sup>239</sup>Pu is less than 5% of the total fissile gram equivalent. Based on packaging the wet sludge in 55-gal. drums, none of the MVST sludge would approach the RH-TRU limit of 325 g per canister for the <sup>239</sup>Pu FGE. Estimates for the total weight (Kg) of sludge in a 55 gal. drum, for each MVST sludge sample, are listed in Table 19.

Table 16 Estimates for <sup>239</sup>Pu FGE with the MVST Sludge

Isotope	<sup>239</sup> Pu FGE	W-24	W-25	W-26	W-27	W-28	W-31
	factor	(mg/Kg)	(mg/Kg)	(mg/Kg)	(mg/Kg)	(mg/Kg)	(mg/Kg)
<sup>233</sup> U	0.865	3.58	6.6	25.1	2.52	12.0	10.9
<sup>235</sup> U	0.641	33.2	41.0	51.3	35.7	46.2	121
<sup>239</sup> Pu	1.000	0.84	1.46	0.57	0.29	0.36	1.49
<sup>239</sup> Pu FGE	(mg/Kg)	25.22	33.45	55.16	25.35	40.35	88.48
<sup>239</sup> Pu FGE	in 55 gal. (g)	7.2	9.5	15.8	7.6	11.5	26.5

Estimates for the total <sup>239</sup>Pu equivalent activity (Ci) in a 55-gal. drum for each of the MVST sludge samples are listed in Table 17. The <sup>239</sup>Pu equivalent activity is based on following calculation,

<sup>239</sup>Pu Equivalent Activity = 
$$\sum_{i=1}^{A_i} \frac{A_i}{F_i}$$
 (4)

where A<sub>i</sub> is the activity of radionuclide *i*, and F<sub>i</sub> is the <sup>239</sup>Pu equivalent activity weighting factor for radionuclide *i*. The weighting factors for the major radionuclides found in the MVST sludge are listed in Table 17. As shown in the last row of Table 17, all of the MVST sludge estimates for <sup>239</sup>Pu Equivalent activity would be less than 1 Ci for a 55 gal. drum, which is well below the RH-TRU limits. The MVST sludge is well below the CH-TRU limit of 80 Ci of plutonium equivalent activity for untreated waste in a 55-gal. drum and will not approach the 1000 Ci WAC limit for a RH-TRU canister, which holds three 55-gal. drums.

Table 17 Estimates for <sup>239</sup>Pu Equivalent Activity with the MVST Sludge

Isotope	<sup>239</sup> Pu wt. factor <sup>a</sup>	W-24 (Bq/g)	W-25 (Bq/g)	W-26 (Bq/g)	W-27 (Bq/g)	W-28 (Bq/g)	W-31 (Bq/g)
<sup>233</sup> U	3.9	1600	2800	10000	1000	5200	5200
<sup>238</sup> Pu	1.1	3800	7800	5400	2400	3000	13000
<sup>239</sup> Pu	1.0	1900	3400	1300	670	830	3400
<sup>240</sup> Pu	1.0	870	1800	890	370	600	2200
<sup>241</sup> Pu	52.0	14000	26000	15000	6500	12000	24000
<sup>241</sup> Am	1.0	3900	9300	3900	2800	4600	14000
<sup>244</sup> Cm	1.9	22000	58000	28000	17000	25000	110000
<sup>239</sup> Pu Eqv. (Bq/g)		22382.98	53335.17	28588.50	15350.60	23479.27	91107.79
<sup>239</sup> Pu Eqv. in 55 gal. (Ci)		0.17	0.41	0.22	0.12	0.18	0.74

<sup>&</sup>lt;sup>a</sup> Radionuclide-specific weighting factors for the <sup>239</sup>Pu equivalent activity taken from Appendix A of DOE/WIPP-069, Rev.5

There is concern about the thermal power from the decay heat of the radionuclides present in waste packages prepared for WIPP disposal. These concerns are addressed in Revision 5 of the WIPP WAC, with limits of 40 watts for a TRUPACT-II container for CH-TRU waste, and a limit of 300

watts for a RH-TRU canister. High decay heat is also an indicator for potential problems with hydrogen gas generation. The major radionuclides found in the MVST sludge are listed in Table 18 along with the "Q" values needed to calculate the decay heat for each isotope.

An estimate of the decay heat distribution by radionuclide for the MVST sludge samples are listed in Table 19 along with an estimate for an upper boundary for total decay heat that would be in a 55 gal. drum full of wet sludge. These estimates indicate that the decay heat from MVST sludge is far below any of the WIPP WAC limits for thermal power and should have no impact on packaging requirements. For general interest, the relative percent distributions of the decay heat by radionuclide, beta activity, and alpha activity are listed in Table 20. The distribution of decay heat as a function of MVST tank and radionuclide is illustrated in Fig. 5 for beta decay, and in Fig. 6 for alpha decay. Although <sup>241</sup>Pu is a pure beta emitter, it is included with the other actinides for illustration. It is interesting to note that the beta activity dominates the decay heat output and that the heat from alpha decay is generally less than 10% of the total thermal power.

Table 18 Isotopes that Contribute to the Decay Heat in the MVST Sludge

Isotope	"Q" value	"Q" value	W-24	W-25	W-26	W-27	W-28	W-31
	(W/Ci)	(W/Bq)	(Bq/g)	(Bq/g)	(Bq/g)	(Bq/g)	(Bq/g)	(Bq/g)
<sup>60</sup> Co	1.54E-02	4.16E-13	2.80E+04	2.50E+04	5.80E+04	1.20E+04	4.20E+04	2.20E+04
<sup>90</sup> Sr	1.16E-03	3.14E-14	1.40E+06	3.20E+06	7.10E+05	4.50E+05	7.00E+05	1.10E+07
<sup>90</sup> Y	5.54E-03	1.50E-13	1.40E+06	3.20E+06	7.10E+05	4.50E+05	7.00E+05	1.10E+07
<sup>137</sup> Cs	1.01E-03	2.73E-14	5.30E+05	4.70E+05	8.90E+05	3.90E+05	3.10E+05	4.30E+05
<sup>137m</sup> Ba	3.94E-03	1.06E-13	5.01E+05	4.45E+05	8.42E+05	3.69E+05	2.93E+05	4.07E+05
<sup>152</sup> Eu	7.65E-03	2.07E-13	8.90E+04	7.10E+04	6.40E+05	4.10E+04	8.00E+05	3.00E+04
<sup>154</sup> Eu	9.08E-03	2.45E-13	3.80E+04	3.70E+04	2.90E+04	1.70E+04	2.70E+05	2.00E+04
<sup>155</sup> Eu	7.59E-04	2.05E-14	1.00E+04	8.40E+03	6.30E+04	0.00E+00	7.00E+04	0.00E+00
Total beta (C	i/Kg)		1.08E-01	2.02E-01	1.07E-01	4.69E-02	8.64E-02	6.20E-01
<sup>233</sup> U	2.86E-02	7.72E-13	1.60E+03	2.80E+03	1.00E+04	1.00E+03	5.20E+03	5.20E+03
<sup>238</sup> Pu	3.26E-02	8.81E-13	3.80E+03	7.80E+03	5.40E+03	2.40E+03	3.00E+03	1.30E+04
<sup>239</sup> Pu	3.02E-02	8.17E-13	1.90E+03	3.40E+03	1.30E+03	6.70E+02	8.30E+02	3.40E+03
<sup>240</sup> Pu	3.06E-02	8.26E-13	8.70E+02	1.80E+03	8.90E+02	3.70E+02	6.00E+02	2.20E+03
<sup>241</sup> Am	3.28E-02	8.87E-13	3.90E+03	9.30E+03	3.90E+03	2.80E+03	4.60E+03	1.40E+04
<sup>241</sup> Pu β <sup>-</sup>	3.20E-05	8.65E-16	1.40E+04	2.60E+04	1.50E+04	6.50E+03	1.20E+04	2.40E+04
<sup>244</sup> Cm	3.44E-02	9.29E-13	2.20E+04	5.80E+04	2.80E+04	1.70E+04	2.50E+04	1.10E+05
Total alpha (Ci/Kg)			9.21E-04	2.25E-03	1.34E-03	6.55E-04	1.06E-03	3.99E-03
Total beta in 55 gal. drum (Ci):			30.80	57.05	30.61	14.01	24.55	185.61
Total alpha in 55 gal. drum (Ci):			0.37	0.83	0.50	0.25	0.39	1.39

Table 19 Distribution of Decay Heat in MVST Sludge

Isotope	"Q" value	"Q" value	W-24	W-25	W-26	W-27	W-28	W-31
	(W/Ci)	(W/Bq)	(W/Kg)	(W/Kg)	(W/Kg)	(W/Kg)	(W/Kg)	(W/Kg)
<sup>60</sup> Co	1.54E-02	4.16E-13	1.17E-05	1.04E-05	2.42E-05	5.00E-06	1.75E-05	9.16E-06
<sup>90</sup> Sr	1.16E-03	3.14E-14	4.39E-05	1.00E-04	2.23E-05	1.41E-05	2.19E-05	3.45E-04
<sup>90</sup> Y	5.54E-03	1.50E-13	2.10E-04	4.79E-04	1.06E-04	6.74E-05	1.05E-04	1.65E-03
<sup>137</sup> Cs	1.01E-03	2.73E-14	1.45E-05	1.28E-05	2.43E-05	1.06E-05	8.46E-06	1.17E-05
<sup>137m</sup> Ba	3.94E-03	1.06E-13	5.34E-05	4.73E-05	8.97E-05	3.93E-05	3.12E-05	4.33E-05
<sup>152</sup> Eu	7.65E-03	2.07E-13	1.84E-05	1.47E-05	1.32E-04	8.47E-06	1.65E-04	6.20E-06
<sup>154</sup> Eu	9.08E-03	2.45E-13	9.33E-06	9.08E-06	7.12E-06	4.17E-06	6.63E-05	4.91E-06
<sup>155</sup> Eu	7.59E-04	2.05E-14	2.05E-07	1.72E-07	1.29E-06	0.00E+00	1.44E-06	0.00E+00
<sup>233</sup> U	2.86E-02	7.72E-13	1.24E-06	2.16E-06	7.72E-06	7.72E-07	4.02E-06	4.02E-06
<sup>238</sup> Pu	3.26E-02	8.81E-13	3.35E-06	6.87E-06	4.76E-06	2.11E-06	2.64E-06	1.15E-05
<sup>239</sup> Pu	3.02E-02	8.17E-13	1.55E-06	2.78E-06	1.06E-06	5.48E-07	6.78E-07	2.78E-06
<sup>240</sup> Pu	3.06E-02	8.26E-13	7.19E-07	1.49E-06	7.35E-07	3.06E-07	4.96E-07	1.82E-06
<sup>241</sup> Am	3.28E-02	8.87E-13	3.46E-06	8.25E-06	3.46E-06	2.48E-06	4.08E-06	1.24E-05
<sup>241</sup> Pu β <sup>-</sup>	3.20E-05	8.65E-16	1.21E-08	2.25E-08	1.30E-08	5.62E-09	1.04E-08	2.08E-08
<sup>244</sup> Cm	3.44E-02	9.29E-13	2.04E-05	5.39E-05	2.60E-05	1.58E-05	2.32E-05	1.02E-04
Total (W/Kg)	)		3.92E-04	7.49E-04	4.51E-04	1.71E-04	4.52E-04	2.20E-03
Density	(Kg/L):		1.37	1.36	1.38	1.44	1.37	1.44
Total in 55 ga	al drum (Kg):		285	283	287	300	285	300
Total in 55 g	Total in 55 gal drum (Watt):			0.212	0.130	0.051	0.129	0.660

Table 20 Summary of Relative Decay Heat in MVST Sludge

Isotope	"Q" value	"Q" value	W-24	W-25	W-26	W-27	W-28	W-31
	(W/Ci)	(W/Bq)	(% Watt)					
<sup>60</sup> Co	1.54E-02	4.16E-13	2.98%	1.39%	5.35%	2.92%	3.87%	0.42%
<sup>90</sup> Sr	1.16E-03	3.14E-14	11.20%	13.39%	4.93%	8.25%	4.85%	15.66%
<sup>90</sup> Y	5.54E-03	1.50E-13	53.51%	63.93%	23.57%	39.38%	23.18%	74.80%
<sup>137</sup> Cs	1.01E-03	2.73E-14	3.69%	1.71%	5.39%	6.22%	1.87%	0.53%
<sup>137m</sup> Ba	3.94E-03	1.06E-13	13.63%	6.32%	19.87%	22.96%	6.91%	1.97%
<sup>152</sup> Eu	7.65E-03	2.07E-13	4.70%	1.96%	29.32%	4.95%	36.57%	0.28%
<sup>154</sup> Eu	9.08E-03	2.45E-13	2.38%	1.21%	1.58%	2.44%	14.66%	0.22%
<sup>155</sup> Eu	7.59E-04	2.05E-14	0.05%	0.02%	0.29%	0.00%	0.32%	0.00%
Total beta h	neat (%):		92.15%	89.93%	90.30%	87.13%	92.23%	93.88%
<sup>233</sup> U	2.86E-02	7.72E-13	0.32%	0.29%	1.71%	0.45%	0.89%	0.18%
<sup>238</sup> Pu	3.26E-02	8.81E-13	0.85%	0.92%	1.05%	1.24%	0.58%	0.52%
<sup>239</sup> Pu	3.02E-02	8.17E-13	0.40%	0.37%	0.24%	0.32%	0.15%	0.13%
<sup>240</sup> Pu	3.06E-02	8.26E-13	0.18%	0.20%	0.16%	0.18%	0.11%	0.08%
<sup>241</sup> Am	3.28E-02	8.87E-13	0.88%	1.10%	0.77%	1.45%	0.90%	0.56%
<sup>241</sup> Pu β <sup>-</sup>	3.20E-05	8.65E-16	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%
<sup>244</sup> Cm	3.44E-02	9.29E-13	5.22%	7.19%	5.77%	9.23%	5.14%	4.64%
Total alpha heat (%):			7.85%	10.07%	9.70%	12.87%	7.77%	6.12%



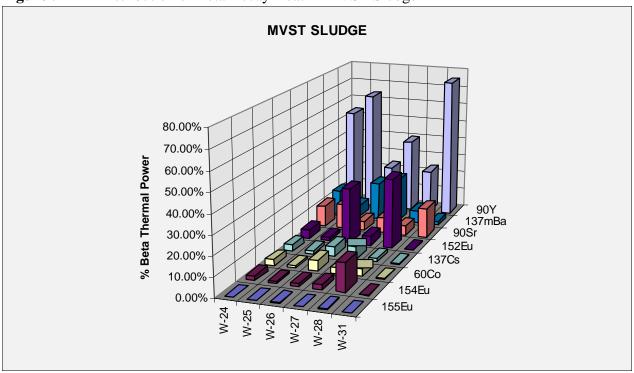
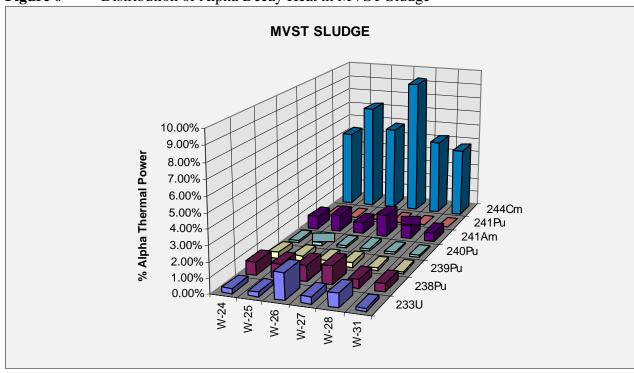


Figure 6 Distribution of Alpha Decay Heat in MVST Sludge

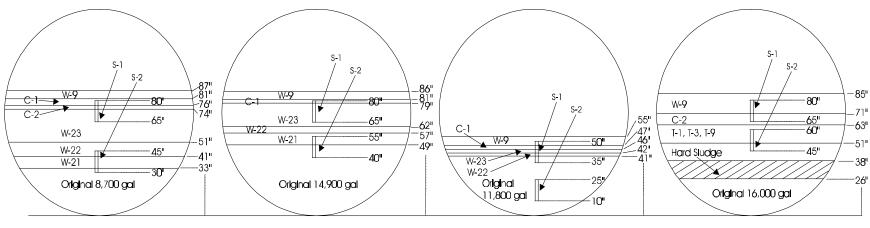


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## **APPENDIX A**

The following diagram graphically illustrates where sludge samples were collected from each MVST tank. Several of the other ORNL waste tanks systems were transferred to the MVST system prior to the sludge sample being collected. This diagram also provides an estimate of which MVST tanks collected these transfers.



Top of G3 Port to Bottom of Tank = 258"

Top of G3 Port to Bottom of Tank = 258"

Top of G3 Port to Bottom of Tank = 255"

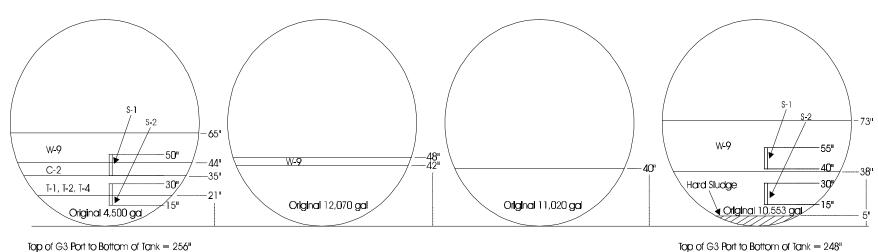
Top of G3 Port to Bottom of Tank = 257"

**W-24** 

**W-25** 

**W-26** 

W-27



Top of G3 Port to Bottom of Tank = 256"

**W-28** 

W-29

W-30

W-31

# **APPENDIX B**

# Radioactive Materials Analytical Laboratory QC Acceptance Criteria for Radioactive Liquid/Solid Waste Samples

Analysis	Method (s) CASD-AM-	Quality Control Check (per batch)	SW-846 Acceptance Criteria (%D, %R, RPD) <sup>c</sup>	RMAL Acceptance Criteria (%D, %R, RPD) <sup>c</sup>
Metals by ICP-AES (inductively coupled plasma atomic emission spectroscopy)	SW846-6010A	high standard calibration verifications (ICV & CCV) <sup>a</sup> calibration blank & checks (ICB & CCB) <sup>b</sup> method blank (sample prep) <sup>c</sup> matrix spike matrix spike duplicate or sample duplicate laboratory control sample (sample prep) <sup>c</sup> serial dilution (if interference suspected) post digestion spike <sup>d</sup>	±5%D ±10%D <3 x IDL <3 x IDL ±20%D ±20 RPD none specified ±10%R ±20%D	±5%D ±10%D <3 x IDL <3 x IDL  23 x IDL  2425%D (liq.), ±30%D (solid) ±20 RPD (liq.), ±30 RPD (solid) ±20%D ±10%R ±25%D (liq.), ±30%D (solid)
Metals by ICP-MS (inductively coupled plasma- mass spectrometry)	SW846-6020	calibration verifications (ICV & CCV) <sup>a</sup> calibration blank & blank checks (CCB) <sup>b</sup> method blank (sample prep) <sup>c</sup> matrix spike matrix spike duplicate or sample duplicate laboratory control sample (sample prep) <sup>c</sup> internal standard post digestion spike <sup>d</sup>	±10%D <3 x IDL none specified none specified ±20 RPD none specified 30-120% R ±10%D	±10%D <3 x IDL <10 x IDL  ±25%D (liq.), ±30%D (solid)  ±20 RPD (liq.), ±30 RPD (solid)  ±20%D  ±30%D  ±20%D
Metals by GFAA (graphite furnace atomic absorption)	SW846-7000A	high standard calibration verifications (ICV & CCV) <sup>a</sup> method blank (sample prep) <sup>c</sup> matrix spike matrix spike duplicate laboratory control sample (sample prep) <sup>c</sup> serial dilution (if interference suspected) post digestion spike <sup>d</sup>	not required ±10%D (ICV), ±20%D (CCV) none specified none specified none specified none specified ±10%R ±15%D	±5%D ±10%D (ICV), ±20%D (CCV) <3 x IDL ±25%D (liq.), ±30%D (solid) ±20 RPD (liq.), ±30 RPD (solid) ±25%D ±10%R ±25%D (liq.), ±30%D (solid)
Mercury by CVAA (cold vapor atomic absorption)	SW846-7471A SW846-7470	instrument blank calibration verification (ICV & CCV) <sup>a</sup> method blank (sample prep) <sup>c</sup> laboratory control sample (sample prep) <sup>c</sup> matrix spike matrix spike duplicate or sample duplicate post digestion spike <sup>d</sup>	none specified	<5 x IDL ±10%D <5 x IDL ±25%D ±25%D (liq.), ±30%D (solid) ±20 RPD (liq.), ±30 RPD (solid) ±25%D (liq.), ±30%D (solid)
Carbon (total organic carbon, total carbon, total inorganic carbon)	SW846-9060	instrument blank calibration verification (ICV & CCV) <sup>a</sup> matrix spike matrix spike duplicate	none specified none specified none specified none specified	<3 x IDL ±10%D (ICV.), ±20%D (CCV) ±25%D (liq.), ±30%D (solid) ±20 RPD (liq.), ±30 RPD (solid)
Anions by Ion Chromatography (IC)	SW846-9056	calibration verification (ICV & CCV) <sup>a</sup> matrix spike sample duplicate	±10%D (ICV), ±5%D (CCV) none specified none specified	±10% D (ICV), ±15% D (CCV) ±25% D ±20 RPD
pH measurement	SW846-9040A SW846-9045B	check standard sample duplicate	none specified	±10%D ±20%D

Analysis	Method (s) CASD-AM-	Quality Control Check (per batch)	SW-846 Acceptance Criteria (%D, %R, RPD) <sup>c</sup>	RMAL Acceptance Criteria (%D, %R, RPD) <sup>e</sup>
Total and dissolved solids (TS & TDS)	EPA600-160.2 EPA600-160.3	sample duplicate check standard	none specified	$\pm 10$ mg/ $10$ mL sample $\pm 10\%$ D
Carbonate and bicarbonate titration	AC-MM-1 003105	sample duplicate check standard	none specified none specified	±20 RPD ±20%D
Gross alpha/beta	EPA-900.0 RML-RA02 RML-RA12	background check calibration verification method blank (optional) <sup>f</sup> sample duplicate matrix spike	none specified none specified none specified none specified none specified	< 3sigma daily change ±10%D evaluated for contamination ±25 RPD (liq.), ±30 RPD (solid) ±25%D (liq.) & ±30%D (solid)
Nuclides by gamma spectrometry	EPA-901.1	background check calibration verification sample duplicate	none specified none specified none specified	< 3sigma daily change ± 10%D ±25%D (liq.) & ±30%D (solid)
Sr-90 determination	RML-RA13 EPA-905.0	method blank (optional) <sup>f</sup> laboratory control sample matrix spike matrix spike duplicate or sample duplicate associated instrument QC	none specified none specified none specified none specified none specified	evaluated for contamination <sup>g</sup> 20%D ±25%D (liq.) & ±30%D (solid) ±25 RPD (liq.), ±30 RPD (solid) see gross alpha/beta criteria
Tc-99 determination	DOE Compendium RP550 RML-RA05	method blank (optional) <sup>f</sup> laboratory control sample matrix spike matrix spike or sample duplicate associated instrument QC	none specified none specified none specified none specified none specified	< 3 x IDL 20%D ±25%D (liq.) & ±30%D (solid) ±25 RPD (liq.), ±30 RPD (solid) see ICP-MS criteria
H-3 determination	EPA-906.0	method blank (optional) <sup>f</sup> laboratory control sample matrix spike matrix spike duplicate or sample duplicate associated instrument QC	none specified none specified none specified none specified none specified	evaluated for contamination <sup>g</sup> 20%D ±25%D (liq.) & ±30%D (solid) ±25 RPD (liq.), ±30 RPD (solid) see gross alpha/beta criteria
Th Determination	EPA-901.1 RML-RA09	method blank (optional) <sup>f</sup> laboratory control sample matrix spike matrix spike duplicate or sample duplicate associated instrument QC	none specified none specified none specified none specified none specified	evaluated for contamination <sup>g</sup> 20%D ±25%D (liq.) & ±30%D (solid) ±25 RPD (liq.), ±30 RPD (solid) see gamma spectrometry criteria
PCBs (polychlorinated- biphenyls)	SW846-8080	calibration verification (ICV & CCV) <sup>a</sup> method blank (sample prep) <sup>c</sup> surrogate standard matrix spike matrix spike duplicate sample duplicate laboratory control sample (sample prep) <sup>c</sup>	refer to method 8080 none specified none specified none specified none specified none specified none specified	to be specified <sup>h</sup> < regulatory limit (2ppm) $\pm$ 50-150%R $\pm$ 50-150%R $\pm$ 50-150%R to be specified <sup>h</sup> to be specified <sup>h</sup>

- a Initial calibration verification (ICV) is typically performed at the beginning of a run to check the calibration and must be independent of the calibration standards. The continuing calibration verification (CCV) must also be independent of the calibration standards, but may be the same standard as the ICV. The CCV is typically analyzed every 10 samples and at the end of the run for metals analysis or every 12 samples for organic analysis.
- b The calibration blank is an instrument blank used in the calibration to initially determine the blank value and therefore used as blank subtraction. The continuing calibration blank (CCB) is also an instrument blank which is analyzed every 10 samples and at the end of the run, but is not used in blank subtraction, but only to monitor instrument contamination.

- c Method blanks and laboratory control samples are only required if a sample preparation is performed before analysis. Sample preparation does not include dilutions or transfers to containers.
- d Post digestion spikes are not necessary if the pre-digestion spike is in control. If this control does not meet the QC acceptance criteria, the post digestion spike should be performed.
- e Acceptance criteria:

%D = % deviation from true value

%R = % recovery of true value

RPD = relative percent difference between two compared values

- f Method blanks for radiochemical analysis are used to monitor cross contamination. However, due to the levels of radioactivity present in samples at the RMAL, the effect of contamination may be insignificant in most cases. Therefore, the requirement to analyze a method blank for radiochemical analysis is optional (i.e. at the discretion of the chemist or supervisor).
- g Acceptance criteria for the method blanks performed for radiochemical analysis varies based upon the level of activity in the samples and the amount of background activity. A qualified chemist reviews the data from method blanks to determine if significant contamination is present.
- h The acceptance criteria for PCB analyses which are not identified in this table, shall be specified at a later date. Currently, the Analytical Methods Group group leader specifies the QC criteria if different from SW846 and if not specified by the sample generator.

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